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Performance Testing of Marine Coatings:
New Test and Evaluation Procedures

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PERFORMANCE TESTING OF MARINE COATINGS: NEW TEST AND EVALUATION PROCEDURES

**NATIONAL SHIPBUILDING
RESEARCH PROGRAM**

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FORWARD

This report was prepared as part of the Marad Administration Contract MA-81-FAC-10011, Task 3-83-3, "Performance Testing of Marine Coatings." The work was co-sponsored by the National Ship Building Research Program through committee SP 3 (formerly Panel 023-1) of the Society of Naval Architects and Marine Engineers. The project was administered by Mr. John Peart, of Avondale Shipyard, and Mr. James Ruecker of National Steel and Shipbuilding Co., (NASSCO). The work was performed by the Steel Structures Painting Council, a non-profit technical association, located in Pittsburgh, PA. The principal investigator was Dr. Bernard R. Appleman; other key SSPC technical personnel were Dr. Joseph A. Bruno, Jr., Dr. Simon K. Boocock, and Mr. Raymond E. F. Weaver. Mr. Walter Radut of W. H. Radut Associates prepared Appendix A of the report.

EXECUTIVE SUMMARY

Because of environmental and health restrictions on conventional marine coatings, there is an urgent need for reliable short-term procedures to evaluate performance and durability of coatings for ship tank exteriors and other exposed surfaces in marine environments. Currently used methods are considered inadequate. Long-term immersion or outdoor tests, though generally reliable, are much too time-consuming for today's regulatory timetable. Commonly used accelerated test methods produce results in a short time frame (1-4 months) but have been shown to give poor reproducibility and poor predication of long-term service performance.

Accordingly, the current project was undertaken by the National Shipbuilding Research Program, with the primary objective to develop test procedures that can better predict performance lifetime and susceptibility to early failures of marine coatings.

The project addressed three key components of a test procedure:

- Procedures to induce coating degradation
- Procedures to evaluate coating degradation
- Procedures to analyze data

Five accelerated tests were utilized to simulate marine immersion and exterior environments:

- Immersion in deionized water at variable temperatures
- Immersion in tap water at high temperature
- Immersion in deionized water under various pressure heads
- Condensation cabinet at various temperatures
- Acidified salt spray

In addition, two aggressive outdoor environments were included to help verify accelerated test data and to establish trends for early failure detection.

A novel quantitative visual examination technique was selected to evaluate the extent of degradation. In this technique, a transparent grid is laid over the surface to be rated, and a count made of the number of grid squares containing a defect. Conventional ASTM visual rating methods for rusting, blistering, and scribe undercutting were also used for comparison and as control techniques.

The framework of the test program was in the application of a new procedure for experimental design and analysis. This technique, known as reliability lifetime analysis, is based on observing, recording, and analyzing the times to degradation of a large number of replicates under different conditions. The technique of reliability analysis provides early information on coatings performance by observing how the number of failures of identically prepared systems varies with the time of the exposure and the severity of the stress. The emphasis of this program, therefore, was in detailed analyses of the performance of a small number of coatings under a variety of special conditions. The coatings included two- and three-coat Mare Island Epoxy, MIL-P-24441, untopcoated and topcoated inorganic zinc, and a marine alkyd.

Conclusions on Accelerated Test Methods

In deionized water, blistering of epoxies generally increased as the temperature increased. However, there was a reversal between 80°C and 90°C after several hundred hours (i.e., higher degree of blistering at 80°C), thus, elevated temperatures can change the mode or mechanism of degradation. Immersion at temperatures above 75°C for epoxies are suspect, even runs conducted at 65°C (commonly used industry method) may not be representative of long-term degradation under ambient immersion conditions.

Deionized water at 90°C has proven to be a more aggressive medium than tap water for promoting blistering. Tap water, however, resulted in a greater degree of rusting. Salt water would be expected to be even more aggressive toward rusting, which is often related to defects in the coating. Thus, immersion in deionized water is recommended for testing of the basic properties of the coating material. Other tests can then be used to determine the capabilities of applying a defect-free film.

Epoxy matings were also immersed in deionized water held at hydrostatic pressures of 0, 15, 65, and 150 psi to determine the effect of the pressure on blistering. Although increased hydrostatic pressure did result in increased degradation of coated specimens, the overall effect of pressure is much more moderate than that of temperature. Based on the experiment on epoxy polyamides, it is recommended that for pressurized immersion testing, pressures in the range of 30 to 50 psig are suitable.

Epoxy, alkyd and inorganic zinc coatings were exposed to variable temperatures in the Cleveland Condensation Cabinet, an apparatus which is normally operated at 40°C. The Cleveland condensing cabinet does not simulate the conditions of immersion. This was demonstrated in the case of the epoxy polyamide which showed extensive rusting in the Cleveland condensation cabinet, while in immersion in deionized water, the major degradation was by blistering. The degradation rate of coatings generally increases as the temperature is increased. For coatings with relatively low glass transition temperatures (T_g s), like alkyds, the system may behave differently at the higher temperature; thus it is also important to examine the film properties at different temperatures. For experiments with a conventional condensation cabinet, a maximum temperature of 60°C is recommended.

Untopcoated inorganic zincs were exposed to acetic acid modified salt spray to simulate the effect of reduced pH. The use of acetic acid in salt fog does have an apparent accelerating effect on consuming zinc. This is attributed to the reaction of acid with zinc on the surface. However, the pattern of rusting formed may be different from that observed in conventional salt spray and in exterior exposure. In addition, it has proven difficult to maintain constant spray and collection conditions in the cabinet in the presence of acetic acid.

Two and three-coat epoxy systems and inorganic zinc with and without a vinyl topcoat were exposed to a severe marine environment (Kure Beach, NC) and industrial (Steubenville, Ohio) environment. For the epoxy systems, the major defect has been scribe undercutting. This phenomenon started in as little as three months at Kure Beach, and overall the undercutting tends to be linear with time. These data provide an excellent example of the value of multiple replicates in establishing and projecting early indications of failure. The technique was used to

project times to failure by undercutting at the industrial site, for which greatly reduced degradation rates were observed.

For coatings designed for exterior exposure, testing conducted on outdoor racks is still considered the most reliable evaluation method. Data from Kure Beach and an industrial site demonstrate that use of good statistical methods and sufficient replicates yields significant data relatively early on coating performance. This corroborates recent work compiled by SSPC in the PACE (Performance of Alternate Coatings in the Environment) study. Thus, any accelerated testing program for exterior exposure should include a series placed in an aggressive outdoor site.

Conclusions on Statistical and Evaluation Methods

The quantitative visual evaluation (QVE or grid) method has proven to be very useful and practical. This technique requires additional rating time, often 2 or three times that for rating using traditional methods. However, the additional information is extremely valuable. It allows more precise comparison of performance among two or more systems. It also allows one to compute more precise plots depicting performance versus time.

The grid method is recommended as a supplement to conventional ASTM methods. The use of the latter is still suggested, because of researchers' familiarity with those methods, and to establish better degrees of correlation between the two rating schemes. The grid method is particularly suitable for evaluating rusting. Care is required when using the method to evaluate blistering to ensure consistency among raters. It may be advisable to have one person or team conduct grid ratings to minimize scatter of results.

In comparing the performance of two or more coatings or coatings under different conditions, the concept of time to failure is an extremely useful evaluation tool. It can supplement the usual, more conventional method of computing average ratings at a given time. In many instances the time to failure provides a more meaningful correlation to the field applications. For example, one is more likely to examine a tank or other structure for the percentage of the surface that has failed, rather than judge the need for maintenance or repair based on the "average" condition of the surface.

Failure times and ratings of nominally identical coated specimens tend to vary, similar to behavior observed for other engineering materials. The manner in which the failures are distributed (i.e., narrow or broad range, shape of curve) can provide important information about the coating system sensitivity to variables like dry film thickness, cure conditions, etc. Failure distributions also provide a more sensitive means to differentiate two systems than conventional comparative methods.

Because of the high variability in coating system performance, it is important to select an appropriate number of replicates for performance evaluation. This experiment has shown that a minimum of 10 to 15 replicates is required to construct distribution curves. To compute reliable average ratings, a smaller number of replicates may be acceptable. The usual practice of exposing two or three replicates is not a statistically sound approach. A practical compromise to ensure representative average ratings may be to use 5 or 6 replicates.

**PERFORMANCE TESTING OF MARINE COATINGS
NEW TEST AND EVALUATION PROCEDURES**

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I. BACKGROUND AND APPROACH

Both of the predominant methods for performance evaluation of marine paints (i.e., accelerated laboratory testing and long-term field evaluation), have severe disadvantages for the user or developer of protective paints. Accelerated tests produce results in a relatively short period of time, (one to four months), but are primarily valued as screening tests, and do not give valid prediction of the long-term field performance. To conduct a long-term field performance test, however, is very costly and often impractical.

Certain accelerated tests are designed to simulate the renditions of various types of exposures on ships. For some types of structures, e.g., salt water ballast tanks, the field conditions, consisting of alternating wet and dry cycles, can be reasonably well simulated by artificial means. However, for other environments, such as topside or deck exposures, or partial intermittent exposure in atmospheric, splash and immersion zones (i.e., boottopping), it is exceedingly difficult to simulate the natural environment in an accelerated test. Field service testing on ships presents a further problem because of the difficulty of conducting regular evaluations. One must rely on the ship captain's interest in the program or one must follow the dry dock schedule in order to get a subsequent evaluation. The paint experimenter has virtually no control over the time intervals for evaluating the paint and little control over the type of evaluation or qualification of the evaluator. In some cases, early failures may be repainted, eliminating any possible information on the rate of breakdown or failure mechanism

Recent innovative work by the National Institute of Standards and Technology (NIST) on service life prediction of paints for structural steel offers enormous potential for improving the ability to evaluate and predict paint performance in a relatively short period of time. in work to date, NIST has demonstrated that failure rates of certain alkyd and acrylic coatings at ambient temperature under high humidity can be predicted from the incidence of failure at higher temperatures. At each temperature studied, the number of failures versus exposure time (frequency of failure) corresponded fairly closely to a standard mathematical distribution (the Weibull distribution). Thus, it was possible to relate the failures at the lower temperature, which may take up to several months, to the failure times at the higher temperatures, which required approximately 100 to 700 hours.

OBJECTIVE

The ultimate aim of this program was to "develop pre-failure testing regimens that can better predict performance lifetime and susceptibility to early failure of coatings for various ship corrosion areas."

Within that larger objective several specific objectives were established. One objective was to extend the original work done by NIST to include types of stresses which are relevant to marine coatings. These include salt spray, immersion, and condensation. As in the original NIST work, the strategy was to vary the temperature or pressure while holding the other stresses constant. Thus a major portion of the proposed work was based on conventional and modified laboratory testing regimens. The major difference between this and conventional testing, however, was that these tests were run at different temperatures or pressures and

that, for each condition, a minimum of 20 replicate panels were exposed in accordance with the principles of reliability analysis.

Another important objective was to determine the validity of using early failures in outdoor exposures to predict the entire failure distribution (i.e., the long-term field performance). For outdoor exposures, there is relatively little control that the experimenter can exercise over environmental variables. Some control can, however, be obtained on the average temperatures and salinity of the sites by careful selection of the locations of the sites. Because of the large inherent variability, large numbers of test panels were required here.

A third objective was to determine the value and suitability of quantitative empirical methods for evaluating degradation of coatings. It is hypothesized that such a procedure may provide earlier and more accurate assessment of the extent of blistering, rusting, or undercutting.

APPROACH

This study applied the technique of reliability analysis to evaluate and predict the performance of coatings subjected to various artificial and natural exposure environments. The application of reliability analysis to service life prediction has been pioneered by the NIST. The present work was coordinated with the NIST coatings research team. The technique of reliability analysis provides early information on coatings performance by observing how the number of failures of identically prepared systems varies with the time of the exposure and the severity of the stress.

For this study four different coating systems were subjected to three to four temperatures and pressures in one to two accelerated tests, and two outdoor exposures. For each coating system in each environment, 20 or 30 replicate test panels were prepared as required by reliability analysis. The evaluation was made on a frequent basis using quantitative rating techniques developed by SSPC and NIST. In addition, the coatings were evaluated with conventional (ASTM) procedures. The particular coatings and the test environment were selected based on a review of corrosion and coating needs and deficiencies.

II. EXPERIMENTAL

1. SELECTION OF ACCELERATED TESTING REGIMES

The major exposure environments to be simulated were immersion and exposure to salt spray and moisture. The following tests were selected:

- **Immersion in deionized water at variable temperatures.** Deionized water is a more aggressive medium for promoting blistering than either salt water or tap water. The temperatures selected were 50°C, 65°C, 80°C, and 90°C. One important aim was to determine whether the higher temperatures caused any change in the mode of degradation compared to the lower temperatures. It is noted that commonly used immersion temperature for marine coatings is 65°C.
- **Tap wafer immersion at high temperature.** Tap water was selected to determine if it could be used as an alternative to deionized water. The advantage of using tap water would be to allow a less cumbersome experimental arrangement. As shown in the results section, deionized water resulted in a substantially higher degree of blistering and hence was used for the majority of the experiment.
- **Immersion in water under various pressure heads.** This test was developed under a National Shipbuilding Research Program (NSRP) project. The original experiments were conducted in pressurized water. For this project, for consistency with the temperature immersion study, the experiment was carried out in deionized water. The purpose of the pressure is to simulate the pressure head which occurs in a ballast tank. The absolute pressures selected were 15 psi, 40 psi, 80 psi, and 165 psi. As with the temperatures, the pressures were selected higher than would be expected under exposure conditions as an approach to accelerating the effect of pressure.
- **Condensing cabinet at variable temperature.** This is a standard industry test which subjects coated specimens to continuous condensation of water droplets on the surface. The test is normally run at approximately 40°C. In this study, the cabinet was fitted with special insulation to allow temperature to be increased. Temperature studies were 40°C, 50°C, 60°C and 70°C.
- **Acidified salt spray.** The salt spray test is the most widely used test for corrosion resistance of coatings. In the conventional salt spray (per ASTM B-117) the panels are exposed to a neutral (pH of 6-6.5) salt spray at 35°C. This project evaluated the use of salt spray acidified with acetic acid (ASTM B-287). The low pH (3.5) of the spray would simulate marine areas with heavy industrial input. In particular, this test was considered a good approach for accelerating the degradation of inorganic zinc-rich primers, which have extremely long durability records in salt fog cabinets but which are susceptible to reaction with acid.

- **Outdoor exposure.** For validating tests on exterior durability such as salt spray and condensation cabinet testing, a set of panels was also exposed at two outdoor test fence locations. These included a well-known severe marine site (Kure Beach, NC 25-meter lot) and an industrial site characterized by relatively high power plant stack emissions and a low pH of precipitation (acid rain).

2. **SELECTION OF COATINGS**

The goal of the program was to evaluate test procedures, not coatings systems. Consequently, a limited number of coatings were selected, as follows:

1. MI L-P-24441, Formula 150-R66, Mare Island Epoxy, Rule 66 Version
 - a. 2 coats
 - b. 3 coats
2. SSPC-Paint 20, Type Ic, Ethyl silicate Solvent Reducible Inorganic Zinc-Rich Primer
 - a. untopcoated
 - b. topcoated with SSPC-Paint 9, White (or Colored) Vinyl Paint.

One of the most important systems is the epoxy polyamide developed by the Department of Defense (MI L-P-24441). For this study, we selected an epoxy coating system with an exempt solvent system. The exempt solvent version, Type 11, was based on use of "Rule 66" solvents, which are those solvents believed to have a lesser impact on photochemical reactions. Subsequent regulatory efforts have emphasized the reduction of all solvents, and discarded the concept of "exempt" solvents.

3. **INSPECTION AND EVALUATION TECHNIQUES**

The specimens were rated periodically for rusting, blistering and scribe undercutting (for scribed panels). The specimens were removed from the testing chamber for as short a period as possible to minimize any irreversible changes due to the dryout period. During the early stages of the immersion testing, the panels were rated while still in the immersion bath. This was done to allow early ratings at 4, 12, 24 or 48 hours without the need to remove the specimens from the bath, thereby disrupting equilibrium conditions. For ratings that occurred at longer intervals (e.g., one week), the specimens were physically removed from the chambers.

The experiment focused on visual degradation of the matings exposed to the above conditions. Standard methods of evaluation include degree of rusting (ASTM D-610 [equivalent to SSPC-Vis 2]), degree of blistering (ASTM D-714 modified) and scribe undercutting (ASTM D-1 654 modified). These methods were utilized in this study because of their prevalence in the industry and because of their value as control techniques for a new approach.

In addition to these, a new, more quantitative technique, designated as quantitative visual examination (QVE) also known as the "grid method" was also used. In this procedure one places a transparent grid over the surface area and counts the number of squares containing a defect. The grid density used was 36 squares per square inch. By use of this size grid, the results were considered to be close to quantitative estimates of the percentage of the surface covered with

defects. One of the goals of the project was to determine the benefits and possible drawbacks to using such a grid technique.

QVE rating was determined to be particularly useful for rating rusting and blistering on the surface area excluding edges. A grid was also devised for the scribe undercutting, but this grid was not found practical to use in the field. Thus, for undercutting, the rating consisted of measurements of the maximum scribe undercutting over the 4-inch length of the scribe.

4. **PANEL PREPARATIONS**

SSPC has developed an efficient, well-controlled procedure for preparing test specimens, which includes the following features.

- a. Source of Steel: Lots of mill scale bearing 1/4 inch plate steel are purchased from a mill and sheared into 4 in by 6 in or 4 in by 12 in or other appropriate size test specimens.
- b. Identification of Panels: Each panel is assigned a unique 3-letter identification code, which is stamped on the front (at bottom) and on the back (in center) of the test panel.
- c. Surface Preparation: Each test panel is prepared according to the required surface preparation standard. In this experiment the test panels were abrasive blast cleaned to SSPC-SP 5, using medium silica sand as the abrasive.
- d. Application of Coatings: After verification that the coatings are mixed as required, they are applied by conventional air spray to one side of the test panel in a spray booth. When the panel is dry to handle, the back side is painted similarly. Additional coats are applied, following the required intercoat interval.
- e. Coating of Edges; In order to minimize creeping and other edge effects, the edges are dipped one or two times in a suitable paint (e.g., vinyl).
- f. Drying, Curing, and Conditioning; Panels are allowed to dry at ambient laboratory conditions for a minimum of seven days prior to any exposure testing.
- g. Measurement of Dry Film Thickness: When the coatings are dried hard, the DFT is measured using a magnetic gauge at a minimum of five locations on the test panel. An average DFT is then computed. If the average or the individual DFT ratings are considered too far from the mean, the test panels will be discarded. Extra test panels were included in the design to allow for this contingency.

5. SUMMARY OF TEST MATRIX

Table 1 lists the 28 individual laboratory tests that were conducted, including the test number, the test chamber, the test parameters (e.g., temperature, pressure), the duration of the test, the type of coating and the number of test specimens.

Table 2 shows the test series and relevant data that were used for the exterior exposure tests. The exterior exposures were placed on racks at both marine and industrial exposure sites.

Table 1

PARAMETERS FOR ACCELERATED TESTS

<u>TEST NO.</u>	<u>CABINET*</u>	<u>PAINT**</u>	<u>NO.OF COATS</u>	<u>TEMPERATURE</u>	<u>NO.OF PANELS</u>	<u>TOTAL HOURS</u>	<u>COMMENTS</u>
1A	Clev. Cabinet	Inorganic Zn	1	70°C-158°F	20	2568	
1B1		Alkyd	2	70°C-158°F	20	1344	
1B2		Alkyd	2	60°C-140°F	20	1248	
1B3		Alkyd	2	50°C-122°F	20	1368	
1B4		Alkyd	2	40°C-104°F	20	2712	
1C1		Epoxy	2	70°C-158°F	4	1008	
1C2		Epoxy	2	60°C-140°F	20	1008	
1C3		Epoxy	2	50°C-122°F	20	1368	
1C4		Epoxy	2	40°C-104°F	20	2712	
2A	Hot Water	Epoxy	2	90°C-194°F	20	1000	Tap water
2B1	Immersion	Epoxy	2	90°C-194°F	18	1320	Deionized
2B2		Epoxy	2	80°C-176°F	20	648	Deionized
2B3		Epoxy	2	65°C-149°F	20	1920	Deionized
2B4		Epoxy	2	50°C-122°F	20	2352	Deionized
3A1	P/T	Epoxy	2	65°C-149°F	20	1344	150 psig
3A2	Immersion	Epoxy	2	65°C-149°F	27	1344	65 psig
3A3		Epoxy	2	65°C-149°F	14	2016	25 psig
3A4		Epoxy	2	65°C-149°F	14	1944	Atm Press
3B1		Epoxy	3	65°C-149°F	14	2016	25 psig
3B2		Epoxy	3	65°C-149°F	14	1944	Atm Press
4A	Salt/Acetic	Inorganic Zinc	1	35°C-95°F	20	2496	
4AA	Acid fog	Inorganic Zinc	1	35°C-95°F	20	3600	
4AAS		Inorganic Zinc	1	35°C-95°F	4	3552	Scribed
4AAA		Inorganic Zinc	1	35°C-95°F	16	2736	
4AAAS		Inorganic Zinc	1	35°C-95°F	4	2736	Scribed
4B		Epoxy	3	35°C-95°F	20	6768	
4BS		Epoxy	3	35°C-95°F	4	6768	Scribed
4BB		Epoxy	3	35°C-95°F	20	5784	
4BBS		Epoxy	3	35°C-95°F	4	5784	Scribed

- * 1. Cleveland Condensation Cabinet, ASTM D 1748
- 2. Hot Water Immersion
- 3. Pressure/Temperature Immersion
- 4. Salt/ Acetic Acid Fog, ASTM B 287

- * * Inorganic zinc, ethyl silicate solvent reducible (SSPC-Paint 20, Type IC).
- Epoxy - Mare Island Epoxy Polyamide (MIL-P-24441, Type II) conforming to Rule 66.
- Alkyd - Federal Specification TT-P-615, Type II, Basic lead silico chromate oil/alkyd primer.

Table 2

EXTERIOR EXPOSURE PANELS

SITE	COATING SYSTEM	NO. OF PANELS	RATED AT:
Marine ^a	3-coat epoxy ^b	30	3, 6, 12, 24, 36 months
	2-coat epoxy ^b	30	
	inorganic zinc ^c (untopcoated)	30	
	Inorganic zinc ^c /vinyl	30	
Industrial ^d	3-coat epoxy	30	9, 16, 23, 36 months
	2-coat epoxy	30	
	Inorganic zinc (untopcoated)	30	
	Inorganic zinc/vinyl	30	

a Kure Beach, NC 25-meter lot.

b MIL-P-24441, Type II (Rule 66 solvent).

c 2-component, ethyl silicate, solvent-borne.

d Steubenville, OH (roof near power plant).

III. METHOD OF ANALYSIS OF DATA

1. FORMAT AND QUANTITY OF DATA

Each test series consisted of possibly **20** specimens, each of which was rated 5-10 times. For immersion panels, both front and backs were rated separately, resulting in twice as many total ratings.

Each rating (per side) consisted of

- ASTM D-610 rust rating
- ASTM D-714 blistering rating
- Grid rating of rusting
- Grid rating of blistering

The grid area rated consisted of a 5 in by 3 in = 15 square inch area within a 4 in by 6 in panel (1/2 inch on each edge was excluded to avoid edge effects). Each square inch included 36 boxes, resulting in the total grid of 540 boxes (Figure 1). For ease of rating and for possible use in distribution analyses, the rating was performed by counting and recording the number of defect boxes in each of the 15 square inches. The maximum in each square inch was 36. The square inches were labeled A-1 through C-5. Figure 2 illustrates the use of the grid on an epoxy immersion panel.

Thus, for each panel rating, a total of 32 numbers were recorded, 1 ASTM D-610 rating, 1 ASTM D-714 rating, 15 grid blister ratings and 15 grid rust ratings. The first and obvious reduction was to total the number of defect areas from the 15 squares to give a total number of degraded areas. This reduced the number of ratings to four per panel per rating period.

2. RATINGS VERSUS TIME FOR SINGLE AND MULTIPLE PANELS

Each of the four quantities above could be tabulated or graphed as a function of time. For example, Figure 3 shows a typical pattern of the ASTM blister rating and the grid blister rating as a function of time for an epoxy panel immersed in deionized water.

An important part of this project was to determine the need for and benefit of using large numbers of replicates (i.e., fifteen or more). The reason for using a large number of replicates is to observe the variability of the degradation among multiple specimens.

Figure 4 shows the blister rating over time for 20 replicate panels exposed in deionized water at 65°C. Figure 4A shows the ratings in accordance with the modified ASTM D-714 rating, and 4B presents the ratings in accordance with the grid method. For either rating method, at any given time (other than 24 hours) the variation is very large. Figure 4C shows the panels with the highest and lowest number of blister defects.

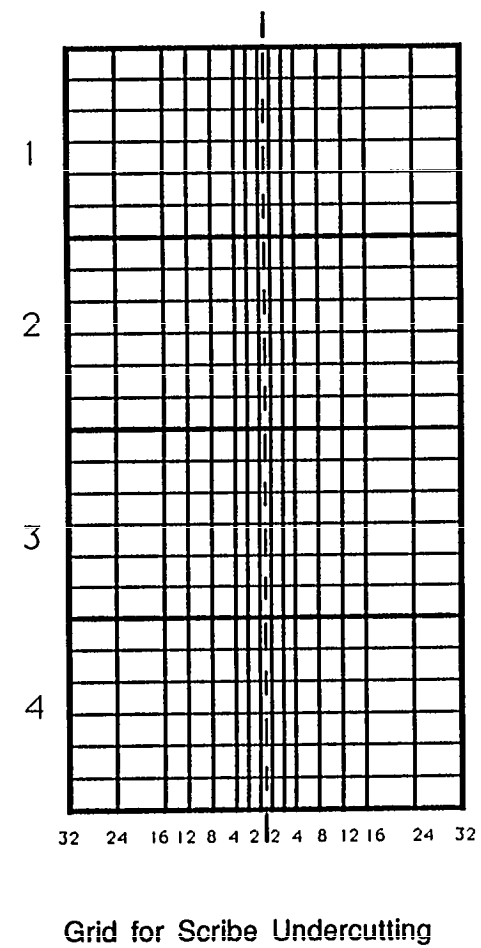
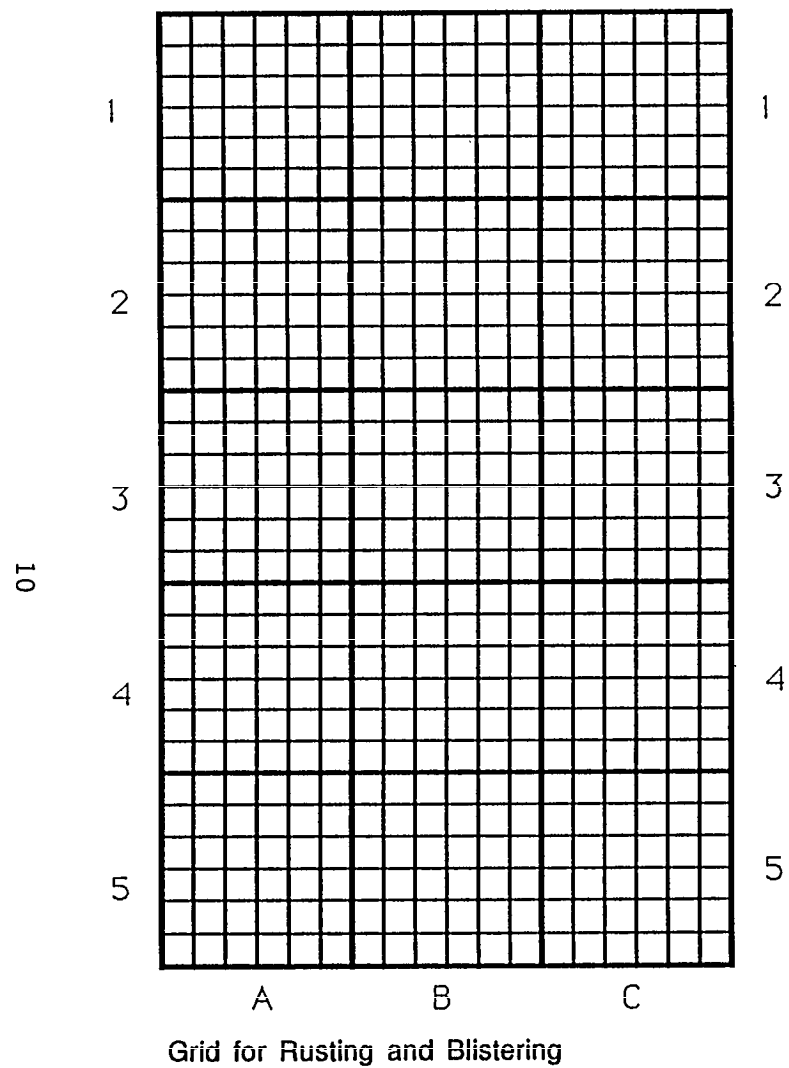
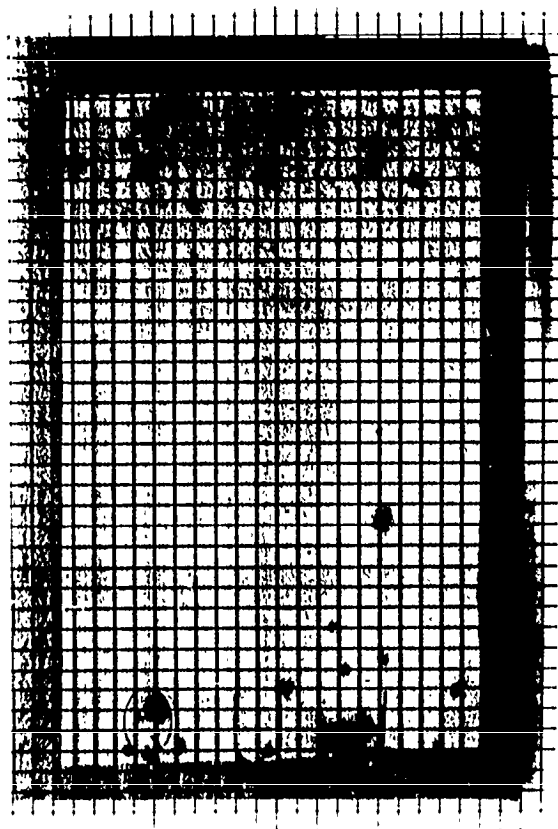
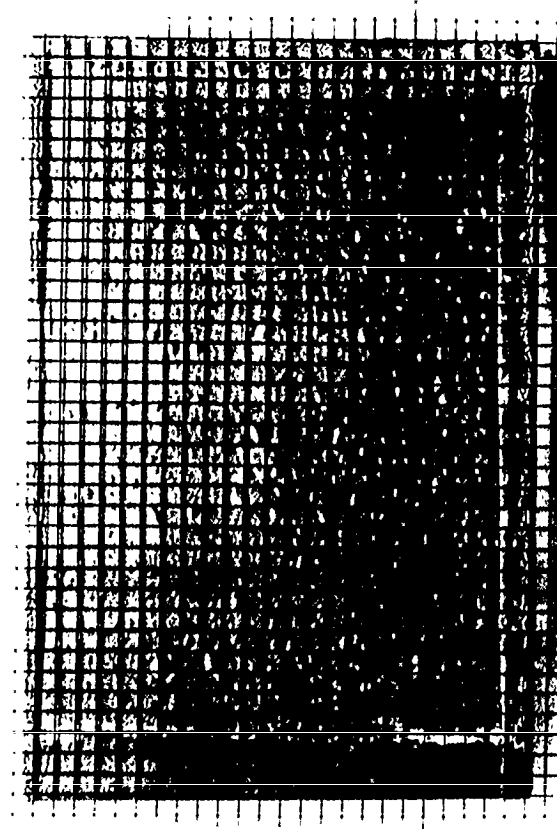


Figure 1. Transparent grids for quantifying measurements of coating defects



Rusting



Blistering

Figure 2 Examples of transparent grid overlay
for rating rust and blister defects

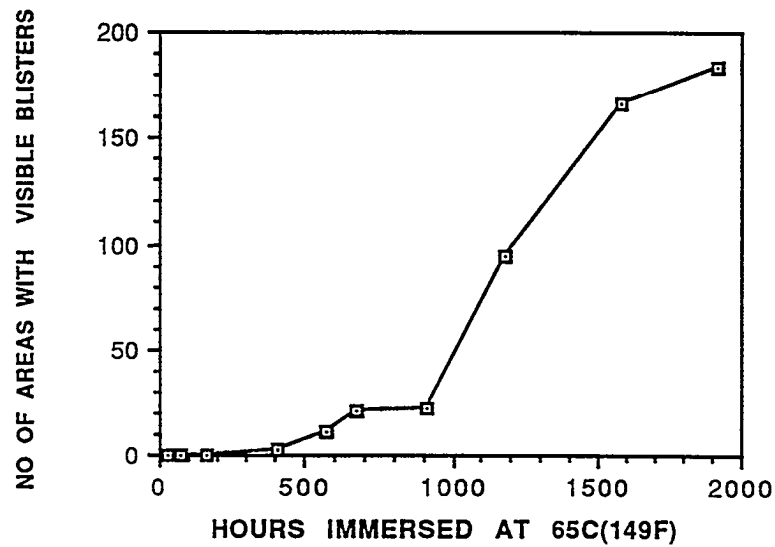
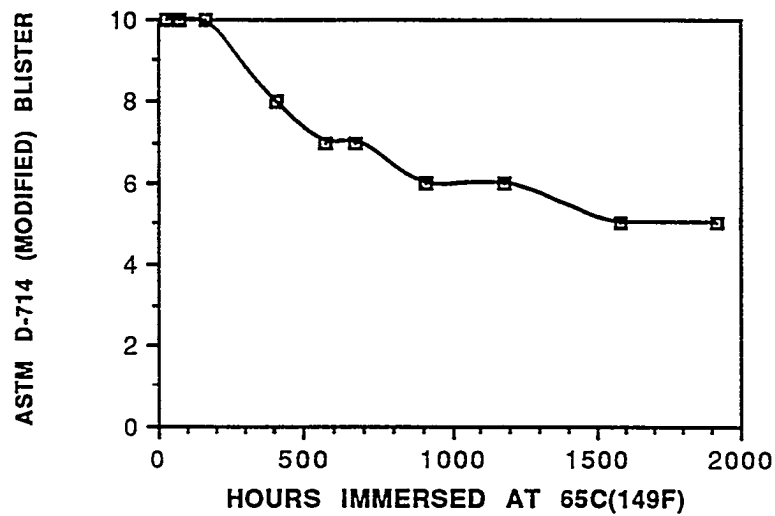


Figure 3 ASTM (upper plot) and grid (lower plot) blister rating over time of single epoxy immersion panel.

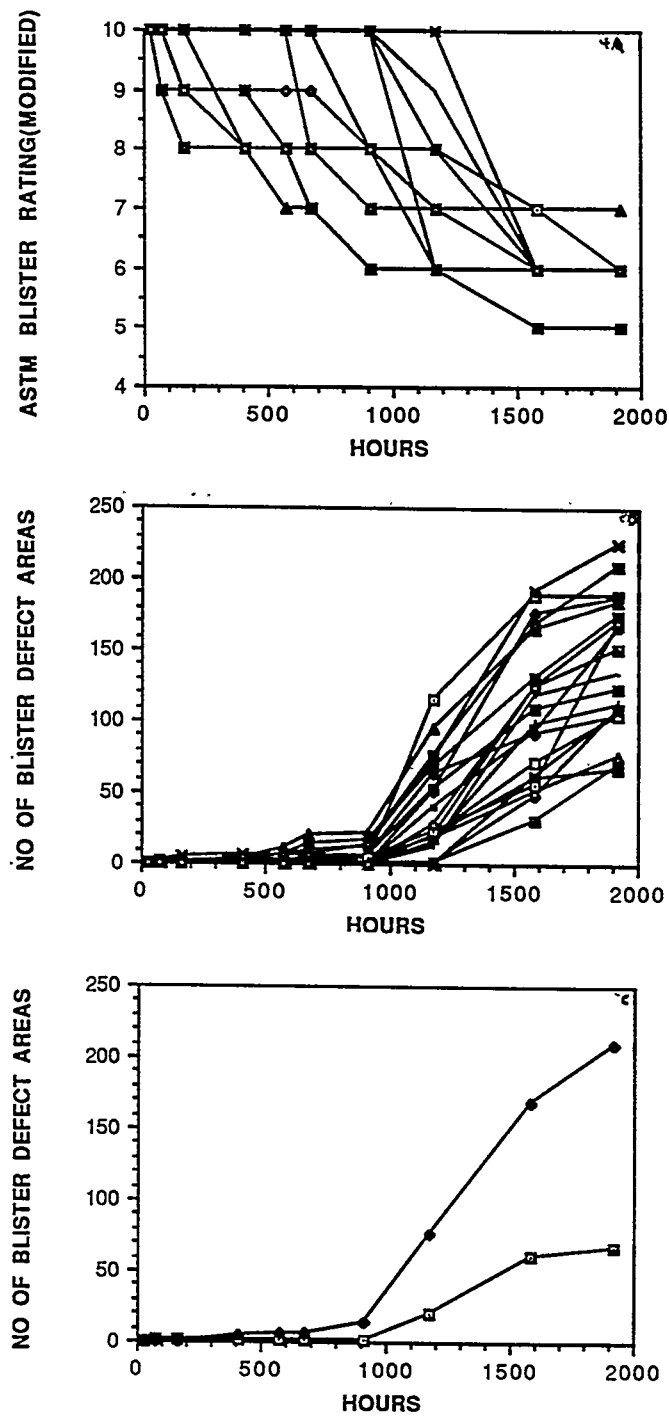


Figure 4 Blister ratings over time for 20 replicate epoxy panels, immersion, 65 °C
 A. ASTM D 714 (modified)
 B. Grid method (quantitative visual evaluation)
 C. Comparison of replicates with highest and lowest number of blister defects

This high degree of variability supports the need to use a large number of replicates, and emphasizes that coating degradation ratings will be distributed at any given time period. Figure 5 is a histogram of the grid ratings at 1200 hours. Because of the wide distribution, standard deviation of the average rating is high relative to the average rating (i.e., coefficient of variation is high).

An informal measure of the variability is a comparison of the average and standard deviation for group of 10 panels each. Four groups selected were the first 10 panels, the second 10 panels, the odd-numbered panels, and the even-numbered panels. The averages and standard deviations for these sets were computed (see Table 3).

3. RATING OF TIME VERSUS TIME TO FAILURE

The most common means of evaluating degradation is by the average rating over time. An alternative approach is to examine the time to reach a prescribed level of performance (e.g., a time to failure). For example, many researchers have used a criterion of a 7 rust rating (per ASTM D-610) as indicative of failure. It is believed, however, that the time to failure is intrinsically more useful than rating at a particular time. The time to failure relates to the need for action (i.e., repainting).

It is also possible to define several different levels of degradation. For example, for the blister ratings shown in Figure 4B, a degradation level can be defined as 10 defect areas on the panel. Thus, for each individual panel, one could interpolate from graphs shown as those in 3B the time until 10 defect areas occurred. The number of hours to reach 10 defects for the 20 replicate panels are as follows, in order of lowest to highest:

540	620	730	790	930	930	930
960	970	990	1010	1020	1030	1030
1040	1050	1130	1240	1250	1290	

The times to failure for a given level of degradation also varied considerably among the 20 replicate panels. The behavior can be represented by a histogram showing the frequency of failure at different time intervals. A failure distribution for 10 defects is shown in Figure 6. Note that the 2 intervals during which the largest number of failures occurred were 800 to 1000 hours and 1000 to 1200 hours. The distribution appears to be somewhat symmetrical but this is not always the case and regular or normal distribution cannot be assumed. Alternate degradation criteria investigated included 3, 30 and 100 defects. A comparison of the distributions for the four different levels of degradation are shown in Figure 7. The higher the degradation criteria, the longer the times to reach that level of degradation.

4. CUMULATIVE FAILURE HISTOGRAMS AND DISTRIBUTIONS

A cumulative failure distribution shows for each point in time the number of failures (or "degradations" that have occurred up to that time. The histogram of Figure 6 can be converted to a cumulative failure histogram as shown in the open bars of Figure 8. The vertical axis has also been changed from number of failures to percent failure. Thus, after 800 hours,

Table 3

SCRIBE UNDERCUTTING RATINGS FOR 2-COAT EPOXY -- KURE BEACH

SCRIBE UNDERCUTTING RATING, 2-COAT EPOXY, KURE BEACH					
	MONTHS				
PANEL	3	6	12	24	36
PXR	2	2	2	4	4
PXT	2	2	2	4	4
PXX	2	3	3	4	4
PXY	2	3	3	4	4
PXZ	2	3	3	6	6
PYA	2	3	3	6	6
PYB	2	4	4	8	10
PYC	1	2	4	10	16
PYD	1	2	10	20	24
PYE	2	2	6	16	20
PYF	2	4	6	14	16
PYG	1	2	10	20	28
PYH	1	2	16	24	32
PYI	2	2	8	16	24
PYJ	2	4	8	16	24
PYK	2	2	8	16	24
PYL	2	4	6	16	24
PYM	1	4	12	16	28
PYN	2	4	4	10	16
PYO	2	4	6	10	12
PYP	1	2	10	16	24
PYR	2	4	4	8	16
PYS	1	2	8	10	20
PYT	2	4	6	12	16
PYU	2	4	6	10	16
PYV	2	4	4	6	6
PYW	1	2	6	24	24
PYX	2	4	4	4	4
PYY	2	4	4	6	8
PYZ	2	4	4	8	10
AVG	1.73	3.07	6.00	11.47	15.67
STD DEV	0.45	0.94	3.22	6.03	8.70
AVG10-1	1.90	3.10	5.70	10.40	14.60
AVG10-2	1.80	3.10	5.10	9.80	13.60
AVG10-3	1.50	3.00	7.20	14.20	18.80
SD10-1	0.65	1.33	4.17	6.93	9.89
SD10-2	0.42	0.99	2.02	4.37	7.53
SD10-3	0.53	0.94	3.26	6.56	9.10
AVG6-1	1.83	2.83	5.50	10.33	13.33
AVG6-2	1.67	3.00	5.33	13.33	17.67
AVG6-3	1.33	2.83	7.83	11.33	17.33
AVG6-4	1.83	3.17	5.83	11.33	15.33
AVG6-5	2.00	3.50	5.50	11.00	14.67
SD6-1	0.41	0.98	3.08	5.57	9.27
SD6-2	0.52	1.10	2.73	7.87	9.33
SD6-3	0.76	1.55	5.72	8.33	12.78
SD6-4	0.79	1.50	3.32	6.97	9.44
SD6-5	0.00	0.84	1.76	4.15	6.65

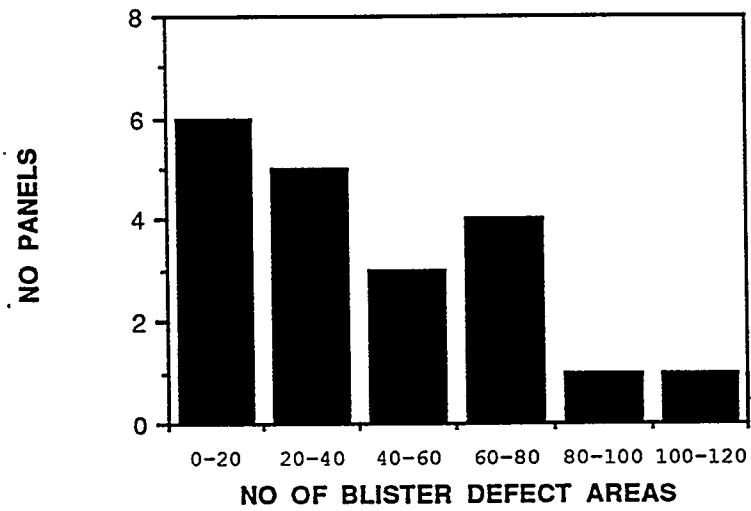


Figure 5 Histogram of blister ratings of epoxy immersion panels at 65 ° C after 1200 hours

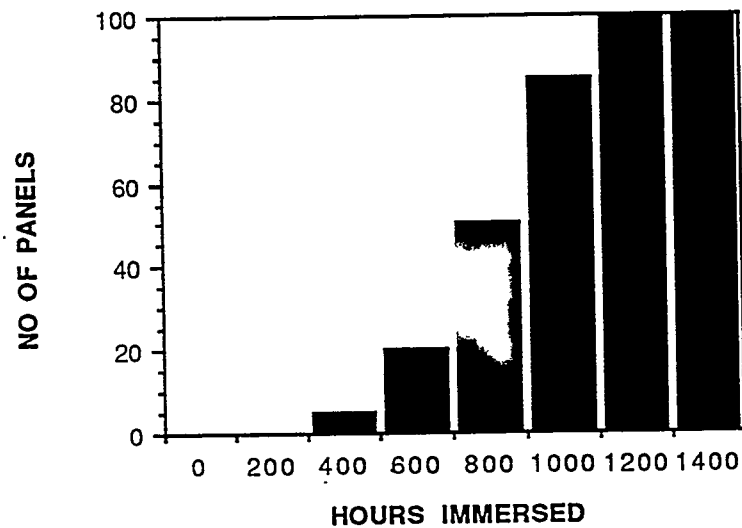


Figure 6 Histogram of time to reach 10 defect areas for 20 replicates for 65 ° C immersion

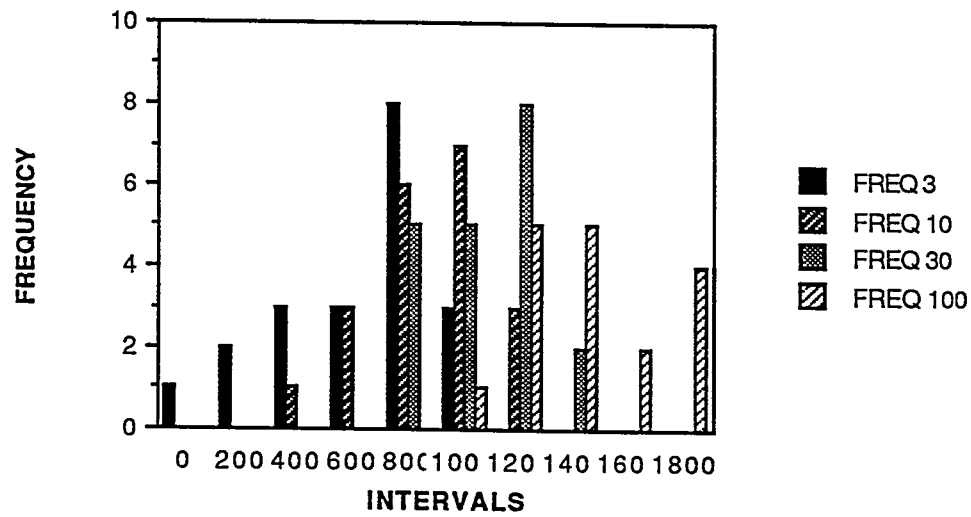


Figure 7 Histograms of times to failure using four different failure criteria for 65° C immersion

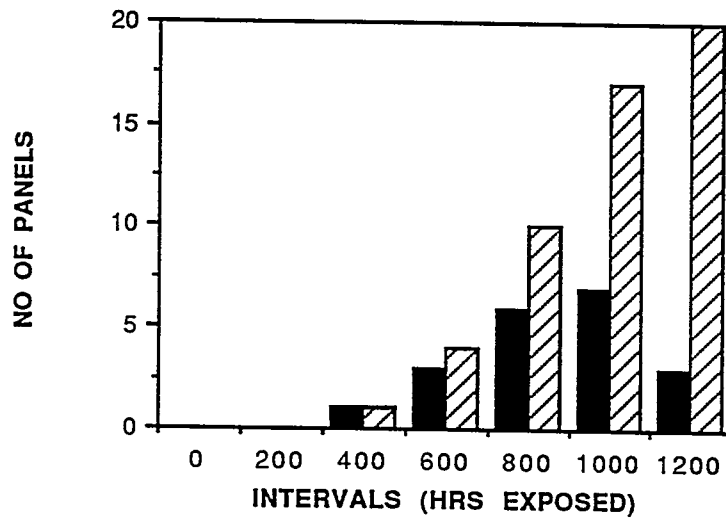


Figure 8 Cumulative failure histograms for times to reach 10 defects for 20 replicates, epoxy 65° C

20 percent (i.e., 4 out of 20) the panels had failed, while after 1000 hours, 50 percent had failed, etc.

A cumulative failure distribution can also be shown on a continuous time axis rather than a histogram, showing the individual failure times for the 20 replicate panels. These points can often be fitted to a mathematical function. Figure 9 shows the failure times for a criterion of ten defects for the epoxy at 65°C fitted to a logarithmic function. Other functions that have been used include exponential, linear, and special mathematical models such as the Weibull distribution.

Figure 10 shows the distribution of the cumulative failure distribution for the four failure criteria for the epoxy at 65°C for blisters. These failures are time to 3 defects, time to 10 defects, time to 30 defects, and time to 100 defects. Each of the four curves is reasonably well separated from the others. For example the median times to failure (i.e., time for 50% of specimens to fail) is 800, 1000, 1150 and 1500 hours, respectively.

The steepness of the curve in Figure 10 indicates the spread of distribution of the failure times. A very steep curve indicates specimens whose failure occurred within a narrow time frame, whereas a flat distribution represents specimens whose failure occurred over a longer time. The shape and distribution of the curve can be described by different mathematical parameters. The discussion of these functions is beyond the scope of this research project.

A physical interpretation of a distribution is as follows: The different test panels represent different portions of a structure that has been painted (e.g., a ballast tank). Thus, within the ballast tank, individual small areas will exhibit slightly smaller paint degradation rates. These differences arise from the variations in the initial surface conditions (e.g., extent of rusting, small metallurgical differences, differences of location within the ballast tank, and differences of the coatings applied (e.g., variations in film thickness, minor differences in application angle, air turbulence positions, etc. Information on the distribution is important. Thus, for example, if 20 percent of a ballast tank is badly rusted, the fact that the “average” rust rating is still above 7 per ASTM D-610 is not very meaningful. Decisions about repair or repainting are often based on the condition of the worst portion of the structure, not the average condition.

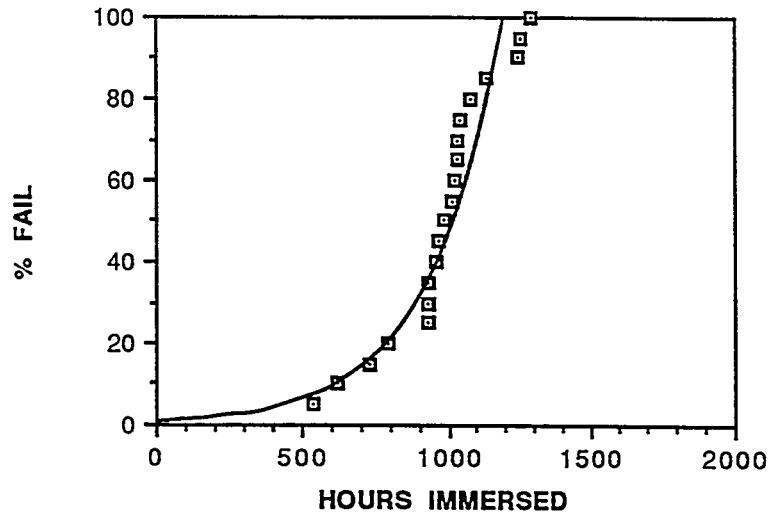


Figure 9 Cumulative failure distribution for times to reach 10 defects for 20 replicates, epoxy 65° C

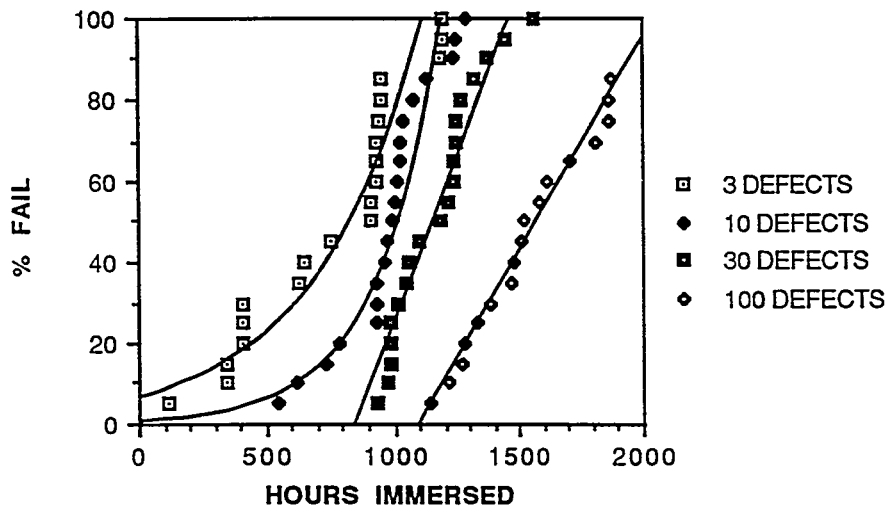


Figure 10 Cumulative failure distribution using four different failure criteria, epoxy 65 °C

IV. DISCUSSION OF RESULTS

1. EFFECT OF TEMPERATURE ON DEGRADATION OF EPOXY IN IMMERSION

Immersion at elevated temperature is the principal means of evaluating coatings designed for immersion services. As described previously, in this project, a standard Navy epoxy was evaluated in deionized water at four separate temperatures: 50°C, 65°C, 80°C, and 90°C. At each temperature, 20 panels were exposed and the backs and fronts rated at regular intervals for rusting and blistering using both the ASTM and grid (QVE) methods.

The principal means of analysis was by computing the times to failure as described in Chapter IV. Alternate failure criteria examined were 3, 10, 30 and 100 blister and rust defects as determined by QVE.

Blistering

The analysis below is based on a criterion of 30 blister defect areas. Based on correlation described in Chapter VI, this most closely approximates a rating of 7 in accordance with the modified ASTM D-714 method.

Figure 11 shows the distribution of failure for three of the four temperatures. At 50°C, only one of the twenty test specimens exhibited as many as 30 blister defect areas. Thus, the distribution of failures would not be meaningful. As expected, the specimens exposed at 65°C had the longest times to failure. However, the distributions at 80°C and 90°C show a reversal of what would be expected: the specimens at 80°C showed a higher rate (shorter time) of failure than did those at 90°C. Also, the distribution curves at these two temperatures have somewhat different shapes, with the failures at 80°C having a steeper (narrower) distribution.

These data suggest that the mechanism for blistering cannot be assumed to be a simple temperature-dependent (Arrhenius) model. Apparently there are other factors that influence the tendency to blister. These could include the rheology of the film, interactions with pigments and film additives, and others. Another possibility is that at 90°C, additional cross-linking of the film occurs, resulting in decreased pitting.

The lack of significant blistering at 50°C further suggests that running experiments at elevated temperatures may not be a good accelerated technique. It seems fairly likely that relatively minor blistering at 50°C occurs by a different mechanism than the substantial blistering at 65°C and the extensive blistering at 80°C.

These experiments were run using deionized water to maximize the driving force for osmosis. It was beyond the scope of the experiment to determine the contents or other parameters of the blisters. It is recommended that additional multi-temperature experiments be conducted using salt water as the medium to determine if the patterns shown here are repeated.

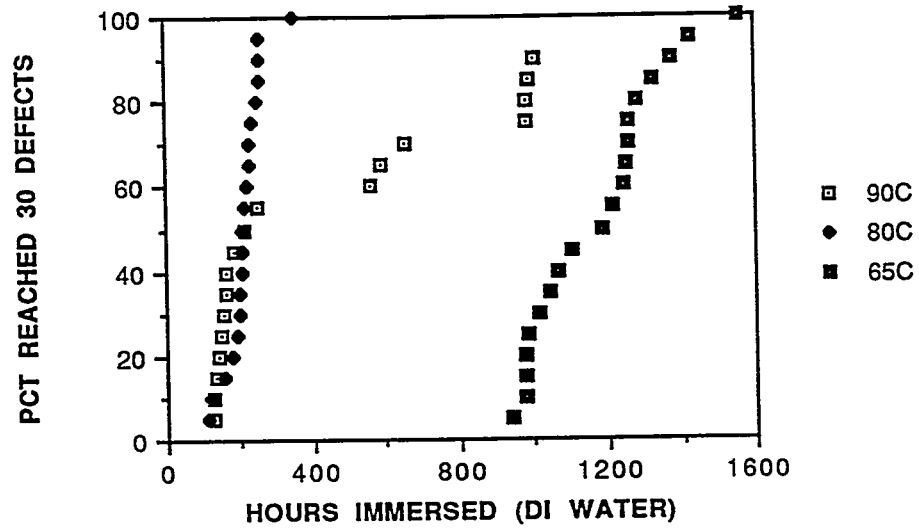


Figure 11 Blister failure distribution of epoxy immersion at three temperatures using failure criterion of 30 defects

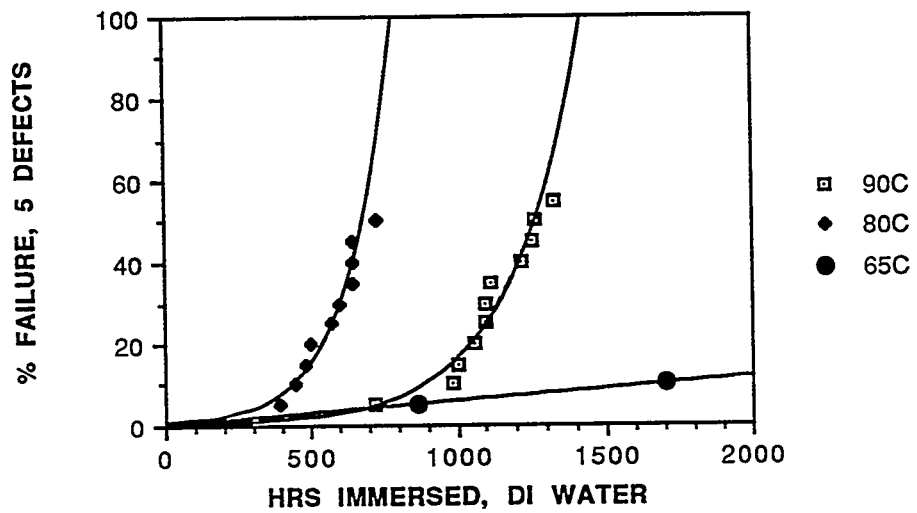


Figure 12 Rust failure distribution of epoxy immersion at three temperatures using 5 defects as failure criterion

Rusting

Overall, the test specimens showed a much lower degree of rusting than blistering. Figure 12 shows the distribution of the times to reach failure based on five defect areas with rust. In this case, the data are shown for three temperatures for the fronts of the panels. The time to rust failure does not appear to depend on the temperature. At 50°C, a number of the panels showed very early signs of rust, but the rust did not progress very rapidly over the duration of the experiment. These rust spots could reflect defects in the applied film. The rust did not propagate because of the relatively unaggressive medium of deionized water.

A general observation was that panels with high degrees of blistering did not have significant rusting.

Epoxy in Tap Water

Figure 13 compares the rusting in deionized water and tap water of epoxy coated panels immersed at 90°C. The rate of degradation by rusting is significantly greater in tap water. This is presumably due to the increased presence of ionic species and oxygen. The panels in tap water exhibited practically no blistering, whereas, as discussed previously, the specimens immersed in deionized water failed exclusively by blistering.

2. EFFECT OF PRESSURE ON DEGRADATION OF EPOXY IN IMMERSION

Increasing the hydrostatic pressure on an immersed coated specimen is an alternate means of driving water into and through the coating. The increased pressure results in a higher rate of diffusion of water through the coating. The accumulation of water at the steel /coating interface will be more rapid than under low-pressure conditions. These conditions are realized at the lower levels of a ship's ballast tank, which may be up to 80 feet deep (at 80 feet, the pressure head is about 35 psi).

In this experiment, the pressure head is simulated by applying compressed air at a given pressure to the closed immersion tank. The four pressures selected were 0, 25, 65, and 150 psig, corresponding to absolute pressures of 15, 40, 80, and 165 psi. The medium was deionized water maintained at a temperature of 65 °C (149°F). The cabinet was repressurized, and the deionized water replaced once a week, at which time specimens were rated.

As described previously, the data were analyzed by computing the time for each specimen to reach a condition of 10 or 30 blister defects. Figure 14 shows the cumulative failure distribution curves for the four pressures using 30 blister defects as failure criterion. The curves demonstrate that the rate of failure increases as the pressure increases. Although the curves are relatively well separated (especially the ones at the higher pressures), the observed differences are not large. The time for 50 percent of the specimens to fail was as follows for the four pressures:

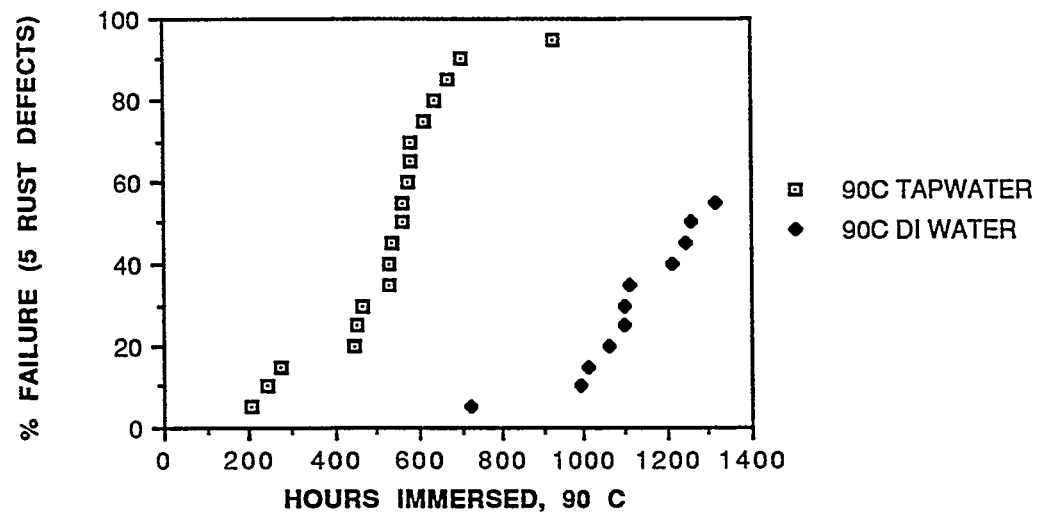


Figure 13 Rust failure distribution, tap versus deionized water, epoxy

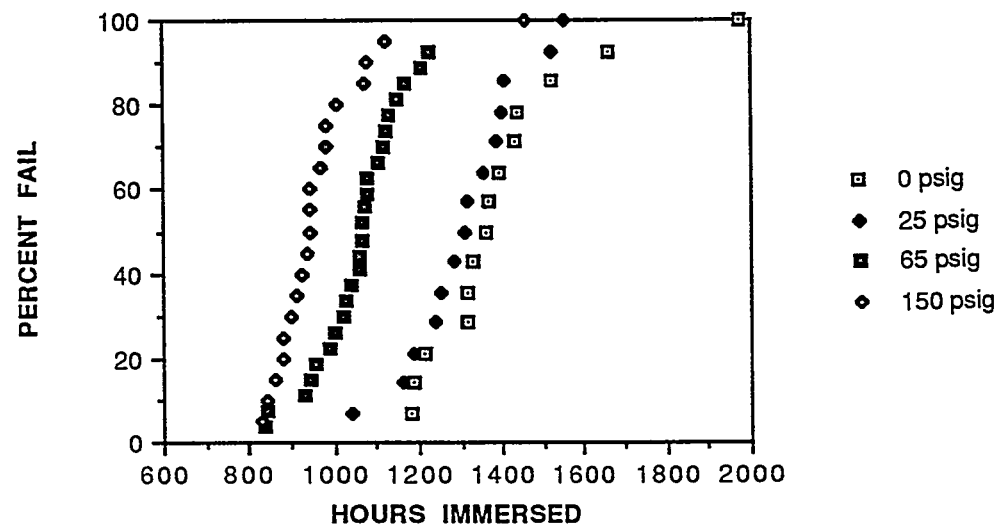


Figure 14 Failure distribution of epoxy immersion at four different pressures, using 30 blister defects as criterion

0 psig:	1375 hours
25 psig:	1300 hours
65 psig:	1050 hours
150 psig:	900 hours

The pressure effect is expected to be dependent on the type of coating system. For poorer barrier systems, or coatings applied over substantially contaminated steel, the differences between the high and low pressure renditions might be much more significant than for the epoxy.

It is also noteworthy that an analysis based on the average ratings of these specimens (rather than the time to failure rating) or using the ASTM D-714 rating (rather than the quantitative grid method) were not able to detect a difference among the pressures. The data thus clearly illustrates the value of the time to failure method of analysis.

In this set, as with the specimens in the temperature series, the backs of the panels exhibit a significantly higher degree of blistering than the front. Analysis of the front showed a similar trend, but the failures occurred at longer times.

Figure 15 compares the failure distribution of identical epoxy series exosed under nominally similar conditions but in different test apparatuses. The conditions are immersion tank at 65°C in deionized water (open to atmosphere) and immersion in pressurized cylinder (closed system) atO psig. The agreement is relatively good.

Rusting

The degree of rusting did not follow the regular pattern observed for blistering. There was no significant difference in the number of rust spots as a function of the pressure. In addition, the degree of rusting did not increase significantly over the length and time of the experiment. For many of the systems, rust appeared within 24 hours and increased only slightly thereafter. The worst specimens accumulated about 30 to 40 rust defect areas, which was substantially less than the amount of blistering observed. For a large majority of the specimens, the rusting of the front of the panel was considerably worse than the rusting of the back of the panel. This is just the opposite of the trend obseived for blistering.

3. EFFECT OF ACID SALT SPRAY ON EPOXY AND INORGANIC ZINC

As described in Chapter III, this test was a variation on the conventional salt spray test. Dilute acetic acid was mixed with the sodium chloride to provide a spray solution at a pH of approximately 3.5. The procedures followed were in accordance with ASTM B-287 (1980) "Method of Acetic Acid/Salt Spray (Fog) Testing." (NOTE: In 1985, this method was incorporated into ASTM G 85, "Standard Practice for Modified Salt Spray (Fog) Testing.")

The coating systems selected for this test were untopcoated inorganic zinc and three-coat epoxy polyamide. For each coating system, 20 replicate specimens were prepared. For the inorganic zinc, the primary objective was to determine whether the low pH of the spray would cause an accelerated consumption of zinc in the coating. The epoxy was included to determine if

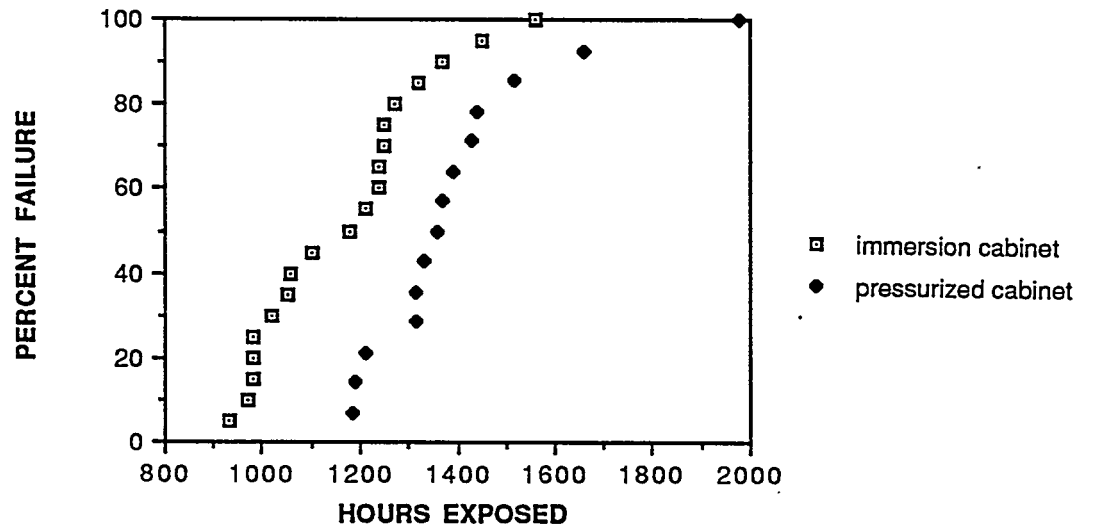


Figure 15 Comparison of failure distribution at 65° C, 0 psi, from two experiments (epoxy)

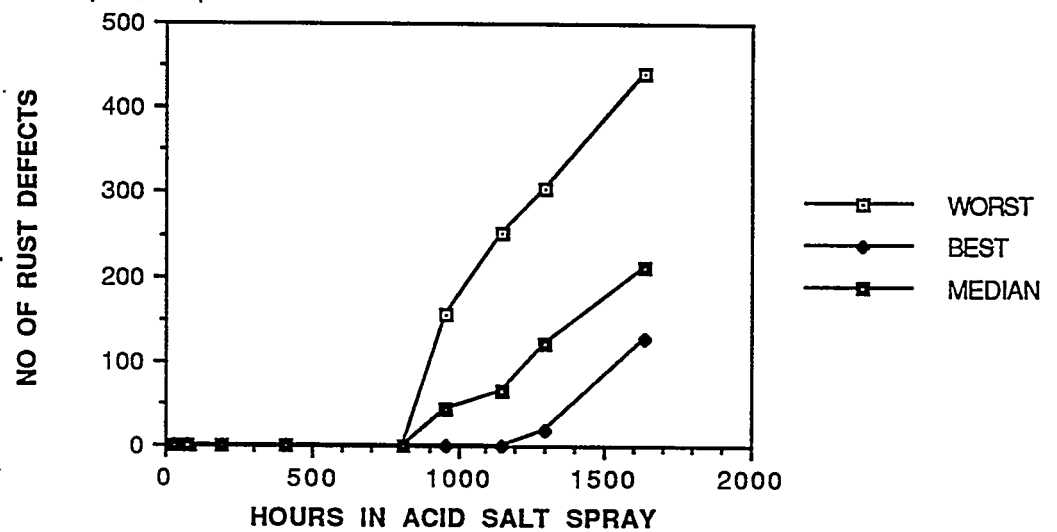


Figure 16 Typical ratings of inorganic zinc rusting versus time in acid salt spray spray test

the low-pH conditions would have an accelerating effect on the degradation of the epoxy coating or affect the rate of undercutting.

It was originally intended to conduct this experiment at several reduced pH levels and also at approximately neutral pH (i.e., standard salt spray testing). However, the laboratory experienced difficulties in maintaining the proper salt collection rate for the acetic acid salt spray runs. Apparently the presence of acetic acid increases the likelihood of the clogging of the nozzle and reduces the collection rate. As a result, the entire experiment was repeated to determine what effect the collection rate had on the degradation rate of the coatings. For the inorganic zinc mating, because of the large difference between the first two runs, a third run was conducted in order to determine if the variation in coating performance between runs was due solely to the collection rate. The results are discussed below.

A. Inorganic Zinc-Rich Coating (Untopcoated)

In the acetic acid salt spray, all the untopcoated inorganic zinc-rich matings eventually failed by rusting of the surface area. Typical rust patterns over time are shown in Figure 16, using the grid rust rating. The specimens typically exhibited excellent rust resistance (O rust defects or ASTM rating of 10) for approximately 800 to 1,000 hours. After that, however, the deterioration was fairly rapid, and by about 1200 hours most of the specimens had substantial quantities of rust. Figure 17 shows the times to failure based on several failure criteria, including 30 rust defect areas, 100 rust defect areas, ASTM D 610 rating of 8 and ASTM D 610 rating of 7.

The inorganic zinc panels exhibited a relatively narrow distribution of failures (i.e., most of the failures occurred within a range of about 200 hours using the grid method and about 300 to 400 hours using the ASTM rating method. This suggests that this coating is relatively insensitive to application variables and to the mechanics of the film formation. A possible explanation is that under the relatively high acidic conditions, the mechanism of degradation was the relatively pure process of reaction and dissolution of zinc with acid.

Figure 18 compares the distributions obtained for the three separate (nominally identical) runs of 20 replicates each. Each of the three runs exhibits a narrow failure distribution pattern, but the runs are distinctly separated on the time axis, indicating a significant difference in the conditions of the test. For example, the time to reach 50 percent failure for these three test conditions was approximately 1100 hours, 1500 hours and 2500 hours for the runs 1, 3, and 2 respectively. Using the criteria of 30 defect areas (not shown on graph), the 50 percent failure times were 875, 1200, and 2000 hours for the same three runs.

The failure times observed are less than those typically observed for inorganic zinc-rich coatings in salt spray testing. Typically, inorganic zinc coatings will give 3000 or more hours in salt fog testing before showing ratings as low as 8.

A control run of the same specimens under standard salt fog conditions corroborates this conclusion. A discussion of the applications and limitations of acetic salt fog testing will be discussed later in the report.

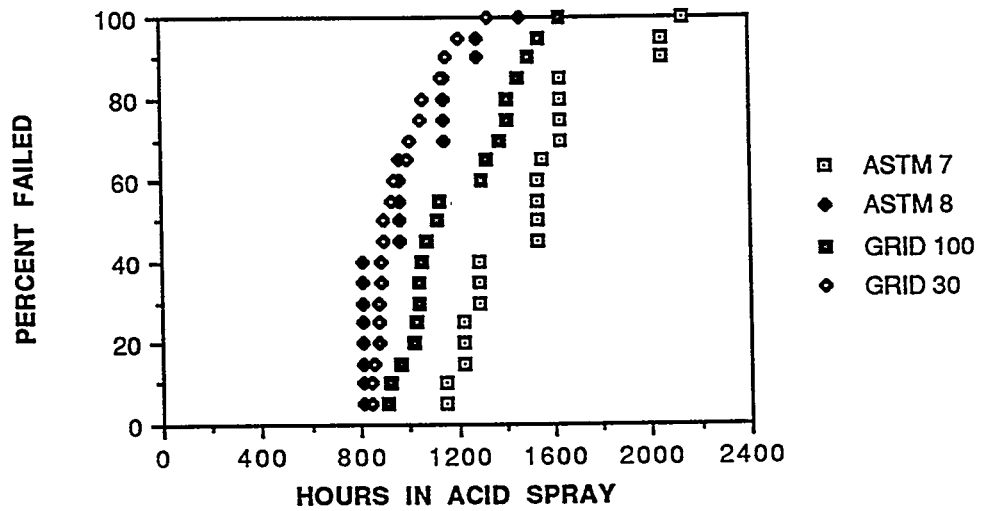


Figure 17 Failure distribution for inorganic zinc in acid salt spray using different failure criteria

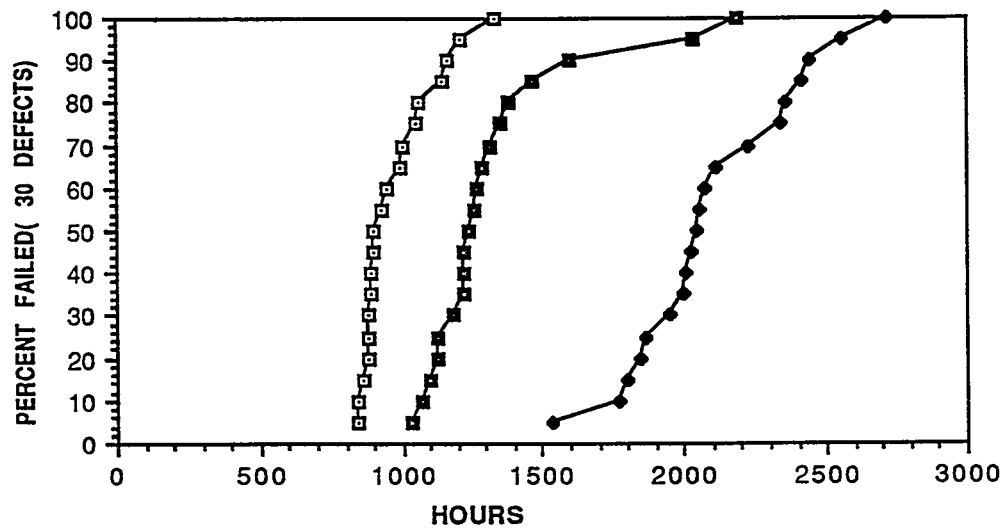


Figure 18 Failure distribution for inorganic zinc in different runs of acid salt fog test

B. Epoxy Polyamide (Three-Coat)

The epoxy coatings degraded almost exclusively by blistering. There was essentially no rust noted for the entire time of the experiment (approximately 6,000 hours). Again, because of variations in the test conditions, two separate runs of 20 replicates each were conducted with the epoxy coatings. In both sets of tests, the epoxies exhibited a blister-free period which ranged from about 1000 to 2000 hours in one test run and 2000 to 2500 in the second. Similar to the case for the inorganic zinc, the two runs had distinctly different degradation ranges.

Some typical degradation patterns of blistering over time are shown in Figure 19. The three plots represent the best, the worst, and the median-performing of the 20 replicates of one run.

Figure 20 shows plots of the failure distribution times for several conditions. For run number 1, the plot shows the distribution based on 10 and 30 grid area defects. For comparison, the failure times are also shown based on 7 rating in the modified ASTM D 714 method. Finally, a plot is shown of the failure times based on ten grid areas for the second epoxy run.

For this test series, the times to reach 10 grid failures is approximately equivalent to the times to reach an ASTM 7 rating. These data also show that the test conditions for the second run were considerably less aggressive and resulted in significantly longer times for blistering to occur. As noted previously, in one of the runs the collection rate of the spray was considerably reduced, which presumably meant that the rate of deposition of the salt was also reduced. Based on the relatively long times-to-failure exhibited by the epoxies in both test runs, it appears that the acetic acid had relatively little effect on the blister tendency of epoxies. It also did not affect the rusting, which was practically non-existent for the epoxy coatings in these tests.

4. EFFECT OF TEMPERATURE ON DEGRADATION OF EPOXY AND ALKYD IN CONDENSATION CABINET

The Cleveland Condensation Cabinet (ASTM D 1748) is commonly used to test moisture resistance of protective coatings. The test panels are placed so that the backside (uncoated) is exposed to ambient laboratory conditions, and the inside (mated) surface is exposed to water vapor at an elevated temperature. Because of heat transfer to the exterior (cooler) side of the panel, the water vapor condenses on the coating. The panels are slanted so that the water runs off the panel, and does not pond on it. Because the water condenses on the panel from water vapor, the water has a very high degree of purity, and thus presents an aggressive medium which can in principle promote osmotic blistering.

For this project, the cabinet was fitted with insulation to allow the panel temperature after condensation to reach 70°C as compared to the normal mode of operation at 4 °C. It was not possible to maintain a constant rate of condensation for the various temperatures at which the tests were conducted (i.e., 40°C, 50°C, and 70°C). The Cleveland condensing cabinet does have an internal thermometer which measures the temperature of the interior surfaces on which condensation occurs, and this was used to adjust the rate of heating to produce the desired temperature.

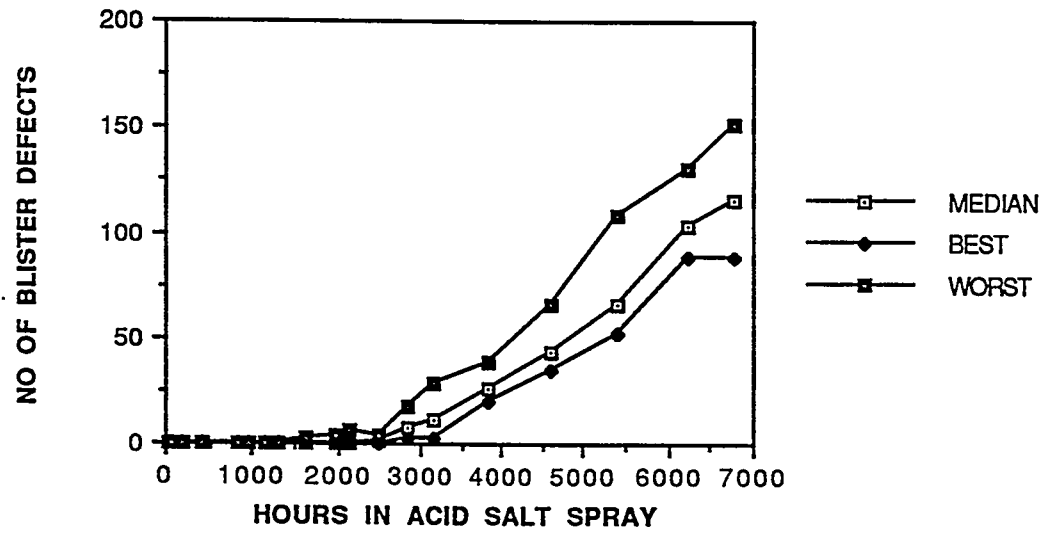


Figure 19 Typical failure patterns of individual epoxy-coated panels in acid salt spray test

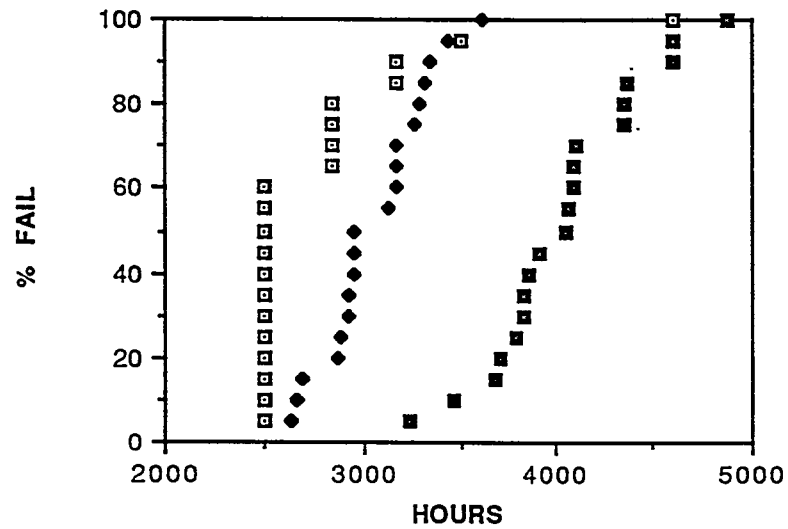


Figure 20 Failure distributions for 3-coat epoxy coatings in different runs of acid salt spray test

A. Epoxy Polyamide (Two-Coat)

Twenty specimens each of the epoxy polyamide were exposed at 40°C, 50°C, and 60°C, and four specimens at 70°C. For the first three temperatures, there was no blistering observed after 1000 hours, at which point the panels were removed. At 70°C, substantial blistering did occur between 500 and 1000 hours. Because there were only 4 panels, it was not possible to construct a failure distribution curve. The final grid ratings for the 4 panels were 42, 38, 55, and 24; each of them had a final ASTM D-714 modified rating of 7.

The specimens at the lower temperatures did show moderate amounts of rusting. These are summarized in Figure 21. Typically, the panels did not show an induction period for rust as was evident for some of the other tests (i.e., acetic salt fog.) Several specimens showed small amounts of rust early in the experiment but did not deteriorate further.

For each of the four temperatures, Figure 21 shows the number of specimens showing different ranges of rust defects. These ranges are 0 to 2 defects, 3 to 5 defects, 6 to 10 defects, and greater than 10 defects. At 70°C, all four of the specimens showed extensive rust degradation with grid ratings of 60, 56, 15, and 53. Moderate quantities of rust were observed after 1000 hours at 60 °C. The most severely degraded specimens showed in the range of 20 to 36 defect areas. Unexpectedly, there were slightly higher levels of rusting at 40°C than at 50°C, but these differences may not be significant. The overall trend then, is that the degree of rusting increases as the temperature increases.

It is also somewhat surprising that the epoxies exhibited rusting rather than blistering, as this test is normally mnsidered to be more aggressive in promoting blistering for the reasons ciied above.

B. Alkyd (Two-Coat)

The alkyds showed a completely opposite behavior from the epoxies in that blistering was by far the predominant mechanism of degradation. Twenty replicates were exposed for about 1000 hours at 40°C, 50°C, 60°C and 70°C. At the three lower temperatures, two to four panels each dropped to an ASTM D 610 rating of 9, but the others remained perfect 10s. At 70°C, all twenty of the panels retained a perfect 10 rust rating for the duration of the experiment.

The alkyd coatings exhibited substantial degradation by blistering at all four temperatures. At 40°C, the coatings were blister-free for between 170 and 330 hours. The blistering that occurred at that time increased substantially up to about 700 hours, but atler that did not show any further increases in number of blisters. Overall the blistering at 40°C was much less than at the higher temperatures.

At the other temperatures, substantial blistering occured within the first 24 hours of the experiment. In particular, at 60°C, all the panels were covered with blisters at 48 hours. Similar pattern was observed at 70°C, but the extent of blistering was slightly less than at 60 °C. At 50°C, about one-third of the panels remained blister-free for between 70 and 130 hours, but after that degraded rapidly.

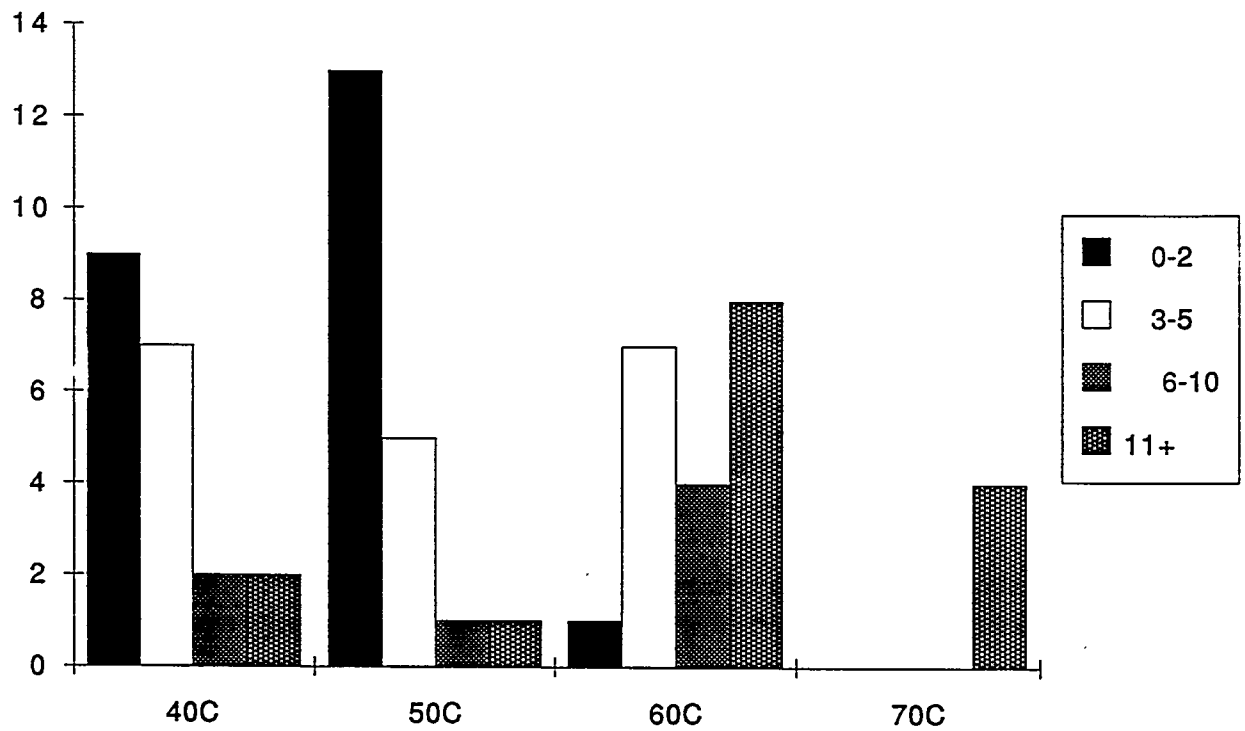


Figure 21 Distribution of rust defects on epoxy in condensation cabinet, 1000 hours

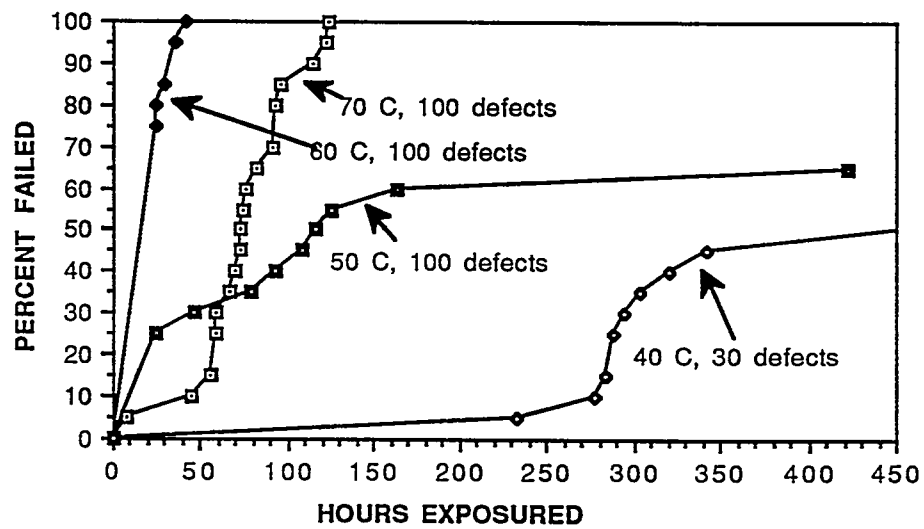


Figure 22 Effect of temperature on alkyd blister degradation distribution in condensation cabinet

Figure 22 shows the failure distributions for the four temperatures. For the three higher temperatures, the criterion is 100 blistered areas, while for the 40 °C panels, the criterion is 30 defect areas.

Here it can be observed that the most rapid failure occurred at 60°C. It is possible that at 70 °C, the softening of the film helped to reduce the rate of blister formation.

C. Inorganic Zinc

A set of 20 replicate inorganic zinc (untopcoated) panels was exposed in the condensing cabinet at 70°C for 2500 hours. Similar to the epoxy polyamide, the zinc-rich coatings showed substantial degradation by rusting but not by blistering. The times to failure (based on 100 grid area defects) were primarily in the range of 2000 to 2300 hours.

5. ANALYSIS OF EXTERIOR EXPOSURES

Test panels were exposed at a marine site (Kure Beach, NC) and an industrial site (Steubenville, OH) to obtain field data on exterior atmospheric exposure. Through the summer of 1989, the panels had been exposed three years. Coatings exposed included two- and three-coat epoxy systems, two-coat alkyd system, one-coat inorganic zinc-rich system, and two-coat inorganic zinchnyl system. Thirty replicates of each coating system at each exposure site were prepared and exposed. The panels were rated for rusting, blistering, and scribe undercutting.

Two- and Three-Coat Epoxy Systems

For the epoxy systems, the major defect has been scribe undercutting. The rating was performed by measuring the greatest distance of undercutting from the center of the scribe. As this is an actual measurement, rather than a comparison to a pictorial, the rating is considered more quantitative than area ratings (e.g., ASTM D 61 O); the rating is still less quantitative than the grid method. The scribe undercutting was considerably more severe at Kure Beach than at the industrial site.

At Kure Beach, the undercutting started in as little as three months, when the first rating was taken (Table 3). As time progressed, the rating of the individual panels deviated significantly (Figure 23). The photographs (Figures 24-25) show the appearance of the epoxies at various stages. As shown in Figure 26, there is a significant difference between the panel with the least and the greatest amount of scribe undercutting for the two-coat system.

Overall, the undercutting tends to be linear over time. Thus the plot of the average undercutting versus time gives an extremely good straight line as shown in Figure 27, comparing two-coat versus three coat epoxy.

Although a grid rating was not performed on the scribe undercutting, times to failure for the individual specimens can be determined by interpolation, using the undercutting ratings. Based on extensive analyses performed in an earlier report, we selected the failure criterion as 8/32 inch (6.3 mm) or 4/32 inch (3.2 mm) undercutting.

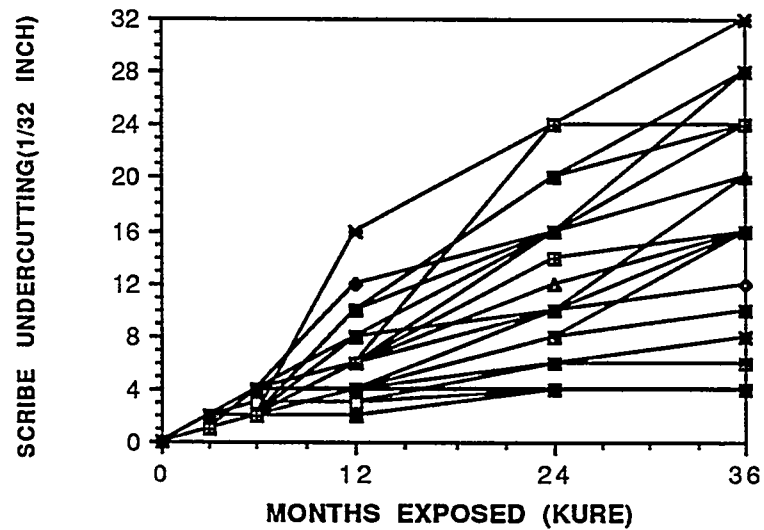


Figure 23 Individual panel scribe ratings, 2-coat epoxy, Kure Beach.



Figure 24 Appearance of 30 replicate scribed epoxy-coated panels after 28 months at Kure Beach

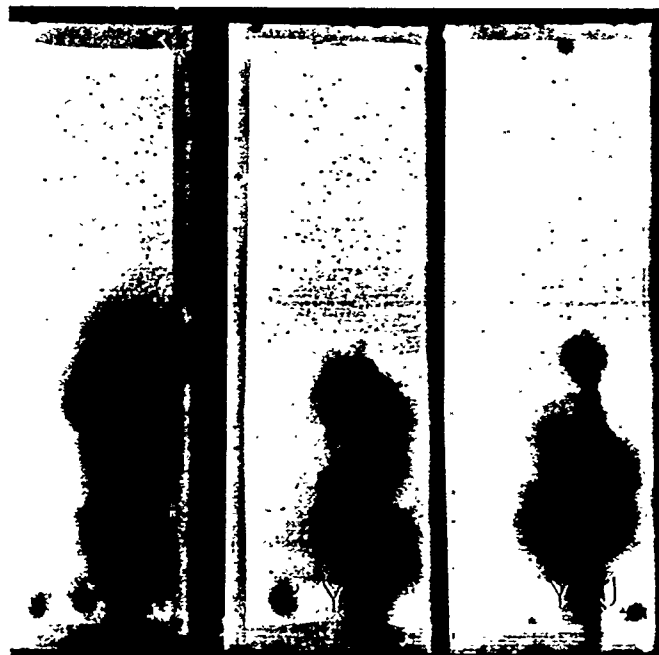


Figure 25 Scribe Undercutting Ratings at two different times for epoxy-coated panels at Kure Beach. 12 months (upper). 28 months (lower).

Figure 28 shows the times to failure for the two-coat and three coat epoxies using a criterion of 8/32 inch. The graphs illustrate that the three-coat epoxy undergoes scribe undercutting failure approximately two months earlier than the two-coat epoxy. This trend is also observed by plotting the average scribe ratings over time for the two- and three-mat epoxies (Figure 27). At the twelve-month point, this trend is more pronounced using the time to failure method than the average scribe undercutting.

Figure 29 compares cumulative failure distribution for a 2-coat epoxy using two different failure criteria, 4/32-inch (3.2 mm) and 8/32-inch (6.4 mm) undercutting. Failures typically occur 4 to 8 months earlier using the more stringent criterion of 4/32-inch.

At the Steubenville (industrial) site, the scribe undercutting was also a linear function of the time exposed. As shown in Figure 30, the two-coat and three-mat epoxy systems gave indistinguishable linear plots. A comparison of the undercutting rate at the marine and industrial sites (Figure 31) demonstrates the markedly increased severity of the marine site.

Based on an extrapolation of the line for the industrial site, one can predict that the average undercutting will reach 4/32 at about 48 months. This is considered to be the first definitive level of degradation. It will be of interest to examine the specimens at 48 months (July, 1990) to see if this prediction is borne out. It will also be of interest to derive a failure distribution curve and compare it to that at the marine site. The difference in degradation rates for these two sites is so enormous that any method can easily reveal this trend. However, such a procedure (i.e., exposing a large number of replicate panels) could be a valuable means of calibrating the severity of different marine and other types of exposure sites.

Other Coatings

The untopcoated inorganic zinc had all "10" ratings for rusting and blistering and "O" ratings for scribe undercutting at both the marine and industrial site at 36 months. The vinyl topcoated inorganic zinc exhibited minor undercutting at the marine site. Thus, after 36 months, seven of the thirty had undercutting of 2/32 inch, with the remaining having undercutting of approximately 1/32 inch. There was also some evidence of undercutting at 24 months. The 48-month rating should provide some interesting results regarding the linear trend of this undercutting.

The alkyd coatings were exposed only at the industrial site. After 36 months, all panels were rated 10's for both blistering and rusting, with very minor scribe undercutting (none more than 1/32 inch).

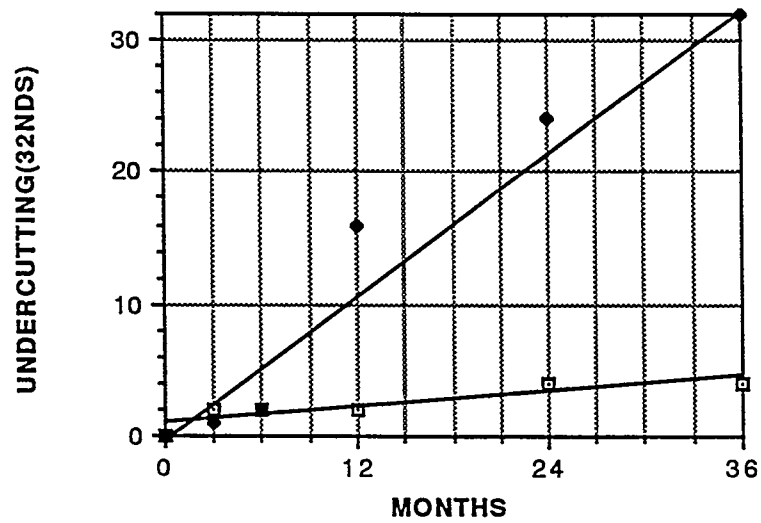


Figure 26 Degradation of panels with greatest and least scribe undercutting, 2-coat epoxy/Kure beach

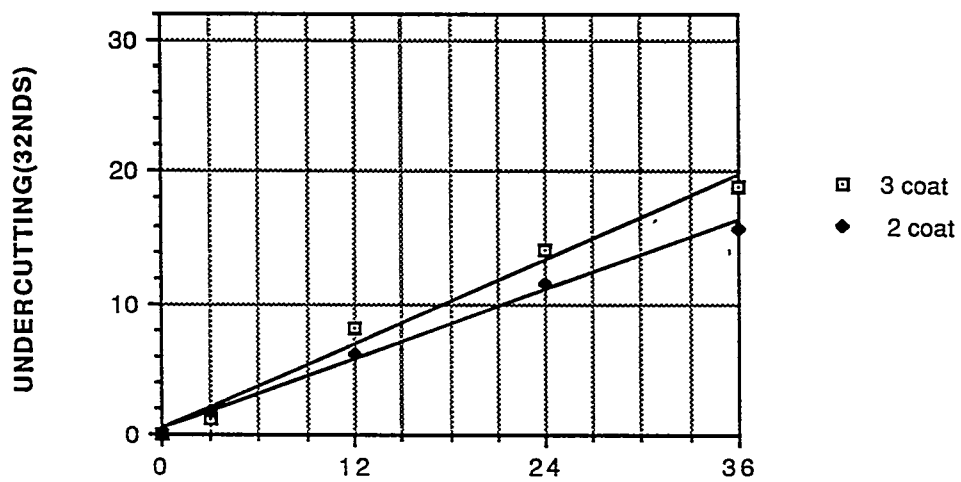


Figure 27 Scribe failure, epoxy, 2- vs. 3-coat (8/32-inch criterion), Kure Beach

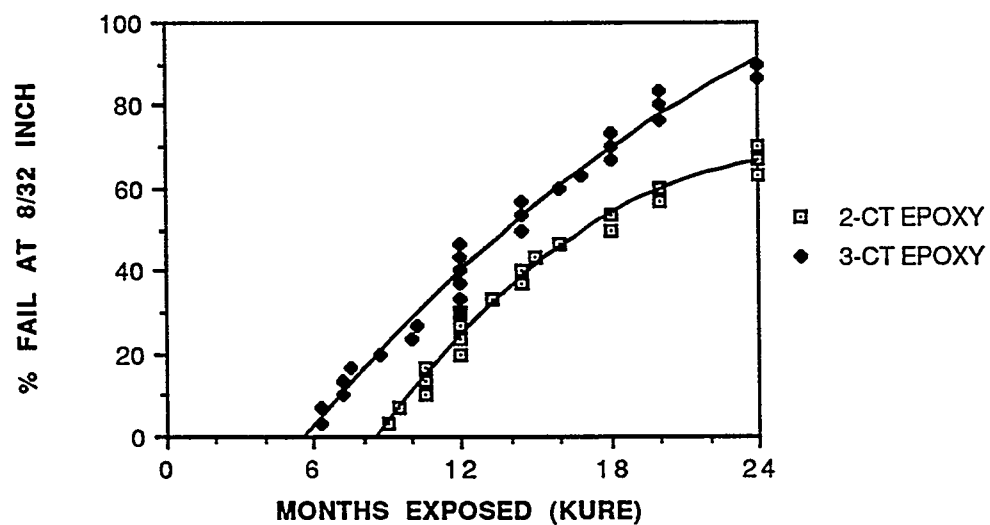


Figure 28 Comparison of cumulative failure of 2- and 3-coat epoxy at Kure Beach

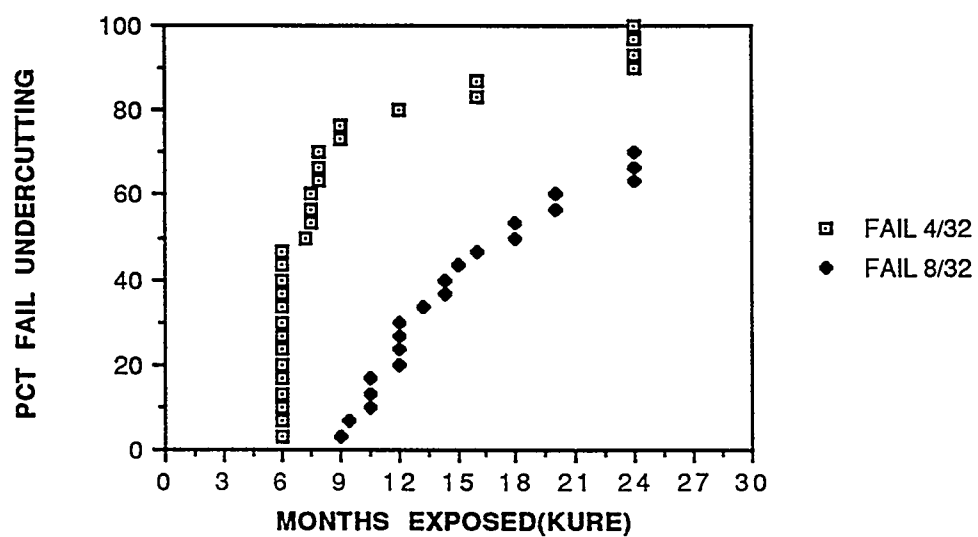


Figure 29 Scribe failures using two criteria, 2-coat epoxy, Kure Beach

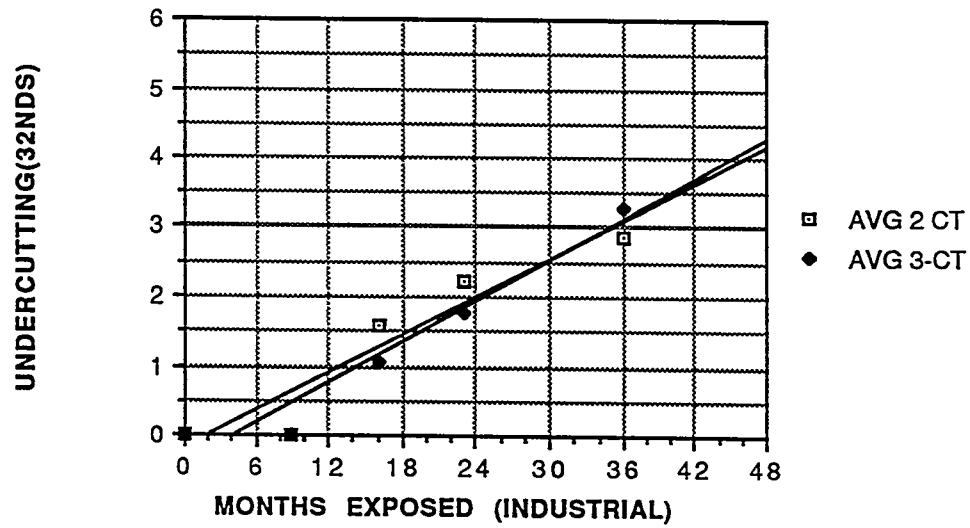


Figure 30 Average scribe undercutting, 2- vs 3-ct epoxy, industrial

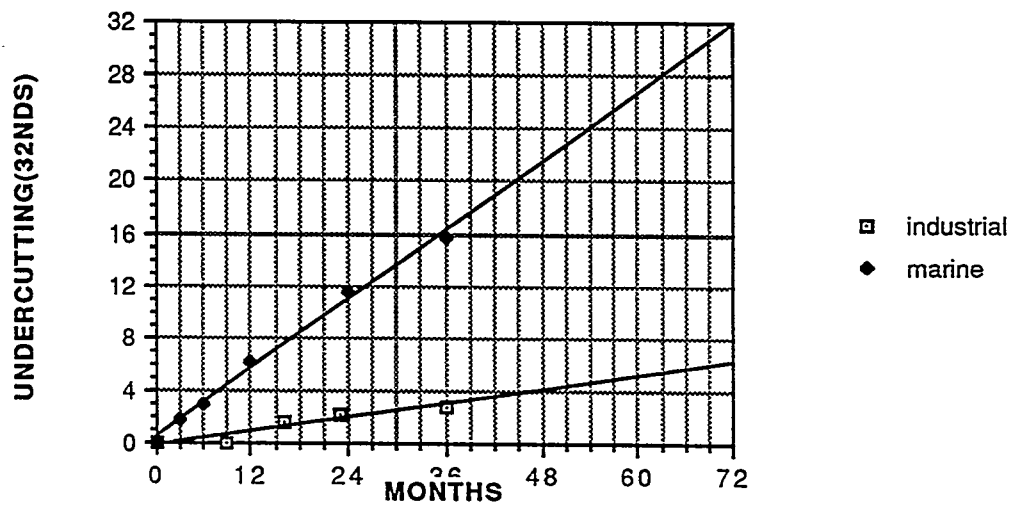


Figure 31 Average scribe undercutting, 2-coat epoxy, marine vs. industrial

V. CONCLUSIONS

1. ASSESSING ACCELERATED TEST METHODS

The project examined several conventional and modified accelerated test methods for marine coatings performance. These included the following:

- High-temperature immersion
- Pressurized high-temperature immersion
- Acetic acid salt spray
- High-temperature condensation cabinet

Other methods, notably cyclic corrosion tests for coatings, are discussed in the appendix.

A. High-Temperature Immersion

The most commonly used test method for evaluating coatings for ballast and fuel tanks and for underwater hulls is immersion. Navy specifications require testing at 180 °F (82 °C) in salt water, deionized water, fuels, and cyclic combinations of these liquids. Other laboratories typically test at 150 to 80 °F (65 to 82 °C) either in salt water, deionized water, or tap water.

In this project, deionized water was selected for several reasons. It is a more aggressive medium for osmotic blistering. It was felt that the effect of temperature would be more easily observed if the presence of other aggressive materials such as salt was eliminated. In addition, recent Navy research studies have indicated that early degradation could be attained using a very low conductivity medium.

For the current experiment, one coating system, a three-coat epoxy polyamide, was utilized. Twenty replicate specimens were immersed at four temperatures: 50 °C, 65 °C, 80 °C, and 90 °C, and blister ratings were obtained periodically using the grid (quantitative) method and ASTM rating schemes. Although increasing the temperature generally increased the rate of degradation, the type of degradation may not be representative of that obtained under service conditions. For the epoxy polyamide, between 80 and 90 °C, there was a reversal (i.e., higher degradation was obtained at 80 °C than at 90 °C). This is tentatively attributed to a change in degradation mechanisms. For example, a slight softening of film (say at 90 °C), could allow trapped moisture or solvent to more easily permeate through the film. Alternatively, an irreversible increase in cross-link density at 90 °C may have so changed the character of the paint film that it became less permeable.

Reducing the immersion temperature to 50 °C (from the more conventional 65 °C) greatly increased the time for degradation to occur. The differences in rates may not be entirely due to the Arrhenius factor (rate vs. temperature). Thus, even experiments run at 65 °C may not be truly representative of long-term service conditions at ambient temperatures. Because of the apparent change in mechanism, it was not possible to derive any rate constants for the degradation as a function of temperature.

B. Pressurized High-Temperature Immersion

Another approach to increasing the rate of degradation is to increase the hydrostatic pressure. This corresponds to the pressure experienced at the bottom of a ballast tank for example. The pressure could increase the stress on the coating and thereby influence the degradation rate.

The experiment was run at a constant temperature of 65 °C, and at pressures of 0, 15, 65, and 165 psig. The specimens exposed at higher temperatures experienced a higher degradation rate than those at lower temperatures. Therefore, pressurized immersion is a means to reduce the evaluation time. The effect, however, was not dramatic; the overall reduction in experimental time was on the order of 40 percent between the highest and lowest pressures (165 to 0 psig).

C. Acetic Acid Salt Spray

In this variation of conventional salt spray testing, acetic acid was added to reduce the pH. An untopcoated inorganic zinc mating and an epoxy polyamide system were exposed. As expected, the presence of the acetic acid accelerated the attack on the inorganic zinc. However, the test conditions proved to be difficult to maintain uniformly. The precision among separate runs was relatively poor. The acetic spray did not have a strong impact on the degradation of the epoxy coatings. Thus this technique may be suited for evaluating systems for their tolerance to acid.

The appearance of the zinc-rich coating in this experiment was slightly different from that observed in standard salt spray testing. Rather than the normal white zinc corrosion product, the surface became covered with a relatively loosely adherent reddish layer of corrosion products. [It is noted that recent results reported in the literature have concluded that the corrosion products obtained in a conventional salt spray test are different from those observed in exterior exposure. Thus the applicability of acid salt spray to exterior exposure seems extremely tenuous.

D. Condensing Cabinet

In this test, we examined the effects of increasing temperature on blistering and rusting of systems exposed to 100% condensing conditions. For alkyds, the blistering generally increased with increasing temperatures. However, there was a slight reversal in that the density of early blisters decreased slightly from 60 °C to 70 °C, although in both cases they were quite extensive. At 70 °C, the alkyd film was very soft, and likely above the glass transition temperature (T_g). Thus, the relief of film stress may have countered the effect of the higher temperature.

Unlike in the acid salt spray cabinet, the epoxy coating exhibited very little blistering in the condensation cabinet experiments. The predominant mechanism of degradation was by rusting.

These results suggest that the conditions for condensation in this cabinet are significantly different than those for immersion or direct solution spray. A possible

explanation is that the coatings were not in a true immersion condition (i.e., were not uniformly wet).

E. Exterior Exposure

Results indicated that epoxy coatings in exterior exposures are susceptible to scribe undercutting. The rate of undercutting is approximately linear with time. The two-coat system gave slightly better undercutting resistance than the three-coat, but the differences were not considered significant. There was a high degree of variability in the degradation rate by scribe undercutting, consequently a large number of replicates is required to provide meaningful analysis.

The average undercutting as a function of time is a useful means to compare performance among coatings and for predicting performance trends of coatings. This was demonstrated by comparing the rates to reach an average undercutting of 4/32 inch and 8/32 inch at Kure Beach. This approach has been used to predict the kinds of failure at the industrial site, which is projected to be at about 48 months. This hypothesis will be checked out at the 48-month rating planned for July 1990.

Inorganic zincs without a topcoat provide excellent rust and undercutting resistance. The application of a vinyl topcoat results in a slight amount of undercutting at the marine site. This is attributed to the vinyl providing a barrier preventing the zinc from sacrificing itself to prevent undercutting. This system, however, undergoes substantially less undercutting than an all-vinyl system based on the results of a prior SSPC project sponsored by the Federal Highway Administration (PACE).

Overall, the undercutting and rusting were much less severe at the industrial site than at the marine site.

2. STATISTICAL METHODS

This project investigated use of new statistical approaches for analyzing coating degradation. The basic approach was reliability analysis, which uses multiple replicates in order to derive distributions of degradation or failure.

The underlying premise is that coating systems are highly complex and variable engineering systems. Thus one needs to analyze degradation in terms of probability and statistics, rather than as defined points or times to failure.

Thus, rather than averaging 2 or 3 test specimens to give a rating at a certain time, it is necessary to expose a relatively large number of specimens (15-30). This permits one to obtain a true picture of the performance under several sets of conditions.

We will now examine the appropriateness of that approach for evaluating coating materials designed for marine applications.

A. Use of Multiple Replicates

For the lab experiments, normally 20 replicate panels were used. For certain immersion series, both backs and fronts were rated, yielding a total of 40 surfaces. However the fronts and backs were analyzed separately to minimize any systematic errors due to orientation or panel preparation.

The data indicated that coating systems do exhibit a range of failure times. The range is highly variable, depending on the system and the exposure. For example, for inorganic zinc matings in acid salt spray, the times to reach a degradation level of 30 occurred within a relatively narrow time band ranging from 850 to about 1200 hours. For the epoxy polyamide system in immersion at 65 °C, the times to achieve 10 blister areas ranged from 500 to 1250 hours. In many instances, not all of the specimens failed (or reached the designated degradation level) within the total exposure period. Normally, it is still possible to construct a distribution curve if there is a minimum of 8 or 10 systems that have failed within the experimental time. This type of behavior is entirely consistent and is commonly observed in reliability analysis of other engineering materials. However it may be surprising for many coating technologists who have not been exposed to this type of analysis. Thus, experiments using 2 to 3 replicate specimens may provide inaccurate assessment of performance.

The variability of individual specimens is illustrated in the analysis of exterior exposure at Kure Beach. Figure 25 shows the range in performance among the best and the worst performing coatings.

In the exterior exposure experiments, 30 replicates were prepared. For each of the rating periods, we have computed the average and standard deviation of the 30 specimens. Also computed is the average and standard deviation of 3 sets of 10 replicates for those same rating periods and 5 sets of 6 replicates (Table 3). These data demonstrate that with 6 or 10 replicates the mean is not substantially different from that observed with 30 replicates, but there is a substantial reduction in the precision.

B. Failure Distribution Curves

There are two basic reasons for using multiple replicates. First, it allows one to compute more precise averages with better standard deviations. For example, by using a large number of replicates one can construct a relatively reliable plot of average scribe undercutting versus time for epoxies exposed at an industrial site. From this, one can project that failure based on 1/8 inch undercutting will occur at about 4 years.

The second reason for using multiple replicates is that they allow one to compute failure distribution curves. Failure curves require a minimum of 10-15 replicates and are consequently more expensive and time-consuming to produce. The advantage is that they allow the experimenter to better differentiate among different systems or conditions. Many of the trends and differences identified in this study would not be evident using conventional averaging techniques, even with 20 replicates. An example is given in Figure 15, comparing the failures at 0 and 25 psig for the pressurized temperature immersion. The average of the times to failure of these two conditions are essentially indistinguishable using average and standard deviation. However, by plotting the results in a distribution curve, one can see clear differentiation in the time to failure pattern.

3. QUANTITATIVE VISUAL EVALUATION

The quantitative visual evaluation (grid) method is an alternate approach to evaluating degradation of coated specimens. The rater superimposes a transparent grid over the area to be evaluated. The rating is obtained by counting the number of grid areas (boxes) which contain a defect.

The grid method is intended to provide information on both the size and frequency of defects. A large defect will typically cover several grid areas and so will result in a higher defect count than a smaller defect. Thus it is a statistical approach to estimating the percent area degraded.

Occasionally a small defect will be located at the border between two grid areas, and result in a count of two defects, correspondingly a large defect may be entirely within the boundaries of a single grid cell. However, statistically, over a large enough area and sufficient replicates, the large defects will result in higher defect count than smaller defects. The statistical probability of producing misleading ratings due to these effects is diminished as the number of replicates increases.

A. Application to Rusting, Blistering, and Undercutting

Early rust defects are normally easy to detect because of the characteristic color and appearance of rust. During more advanced stages of rusting it is important to distinguish between rusting and rust staining, just as in using ASTM D 610.

Small rust defects are more easily detected using the grid method. The grid pattern forces the rater's eye to traverse the entire test area. When using the ASTM D 610, the tendency is to try to take in the entire area in one glance, particularly if it appears to be rust-free. Thus, surfaces which are given a perfect 10 rating upon closer visual inspection may be found to contain 2 to 3 tiny rust spots. These rust spots are valuable in establishing evidence of early rusting.

The grid rating method also makes it easier for the rater to identify edge effects, as this portion of the specimen is clearly demarked. Typically, the area within 1/2 inch of the edge is not included as part of the plane area for rating because it is subject to "creep" from the edge, where corrosion is more likely to occur. It should also be noted that using the grid method, edge corrosion which has progressed into the rating area will be recorded as planar rust. In this case it must be recorded as a note.

Rating of Blisters

Blisters are more difficult to rate than rust because they are deformations of the film rather than actual breaks in the film. The rater must often make a judgment as to whether very minute bulges are actually blisters. This difficulty also occurs using conventional methods of rating blisters (ASTM D 714). The ASTM method has photographs depicting the actual blister size for different numerical ratings. These photos can also be used in the grid method to determine if apparent bulges or defects should be counted as blisters. Thus, if a blister is smaller than the smallest blister shown in the ASTM photos, it should probably not be counted.

For both blister and rust rating, it is useful to have a good source of lighting, preferably one for which the angle of incidence can be varied. This facilitates detection of blisters, particularly on a dark coating or where blisters are just initiating.

Undercutting

The degree of undercutting is typically measured as the maximum penetration of underfilm corrosion along a direction perpendicular to the scribe line. Grid templates were prepared for scribe undercutting. Ratings are performed by counting the number of squares (grid areas) in which undercutting has initiated. The data can also be recorded as a histogram of the undercutting penetration for each linear increment (e.g., 1/8 inch along a scribe). In this project, grid ratings of scribe undercutting were NOT obtained. The conventional method of measuring maximum undercutting is already a direct measurement (in millimeters or inches). Thus, it is already a form of quantitative measurement. In the future it will be instructive to compare the conventional undercutting rating with a grid method.

Time to Perform Grid Ratings

The grid ratings definitely require more time, particularly in advanced stages of corrosion and blistering. In the early stages, there is a slight added time to scan the boxes and record the number of defects. This is estimated to require an additional 2 to 3 minutes per specimen, including recording time. A typical ASTM D 610 or D 714 rating might take 30 seconds, thus a total time would be perhaps 3 to 5 minutes, depending on the severity of degradation.

To reduce the time for advanced stages of degradation, one could adopt a procedure to cease counting at (say) 100 defects. At this point, the specimen is normally well beyond what is normally defined as a failure (i.e., ASTM D 610 rating of 7 or less or ASTM D 714, modified rating of 6 or less).

The average time to do the rating, however, is normally a small portion of the total cost of the experiment. The major costs are in the design, procurement of materials, preparation and application of film, dry film thickness measurement, and equipment setup and operations. An extra few minutes in conducting the rating may well be worthwhile if it results in obtaining of more complete and accurate data on the performance of the coating.

B. Correlations Between Grid and ASTM Rating Methods

Figure 32 shows the correspondence between ASTM D 610 rust ratings and grid ratings for various panels rated at different periods. Panels which received a given ASTM rust grade (e.g., 8) did not all necessarily receive the same grid rating. For example, panels which were rated ASTM 8 received grid ratings ranging from approximately 15 to 100 defects. Thus, a direct 1:1 correspondence could not be established. In Figure 33, the ASTM ratings are plotted against the logarithm of the average grid rating. These data can be fitted to a straight line with a correlation factor of 0.93. This correlation is not surprising, because the ASTM D 610 rating is a logarithmic scale. Recall that ASTM rating of 10 corresponds to 0 percent rust, 9 corresponds to 0.03 percent rust, 8 to 0.1 rust, 7 to 0.3 rust, 6 to 1 Yo rust, etc. The line begins to deviate at rust ratings below 7. As indicated, this represents an average of greater than 100 defect areas, a condition which is normally considered failure.

From Figure 34, it is seen that the spread of grid ratings for a given ASTM rating is considerably greater for blister ratings than for rust ratings. For example, panels which receive a 4 blister rating (using modified ASTM D 714 method) had blister defect counts ranging from 60 to 400. By plotting the average number of defect areas on a log scale versus the ASTM blister rating, one obtains a reasonably good straight line (Figure 35). The slope of the correlation line, however, varies significantly with exposure site, as shown in Figure 31.

At higher temperatures, the panels exhibited a large number of very small blisters. When evaluating using ASTM D 714, the rater does not normally consider these as defects. Thus, it is important to establish a minimum blister size when using the grid rating method.

The small blisters are also sometimes difficult to detect, requiring a light, as discussed previously. Thus, the grid rating of blisters may require a higher degree of operator skill. Based on the above, it would not be possible to obtain a uniform correlation factor applicable to all exposure conditions (Figure 36).

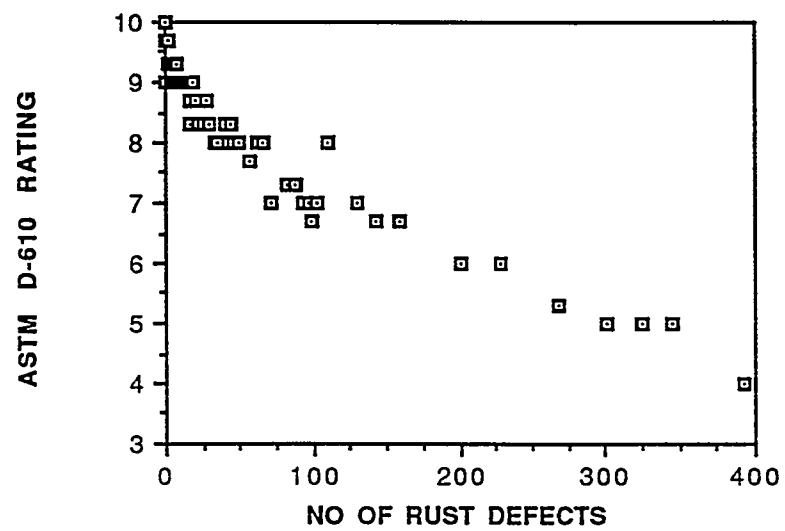


Figure 32 Comparison of ASTM blister ratings average number of blisters, epoxy, 65° C

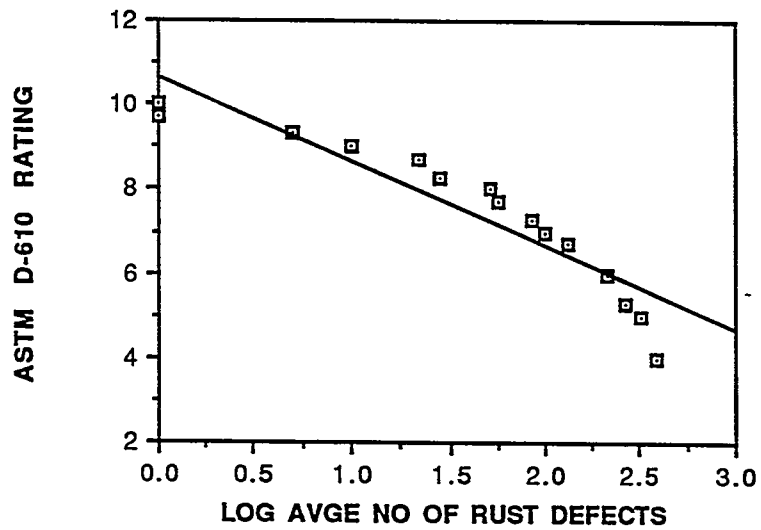


Figure 33 Linear correlation of ASTM blister ratings with log of average number of blisters, epoxy, 65 °C

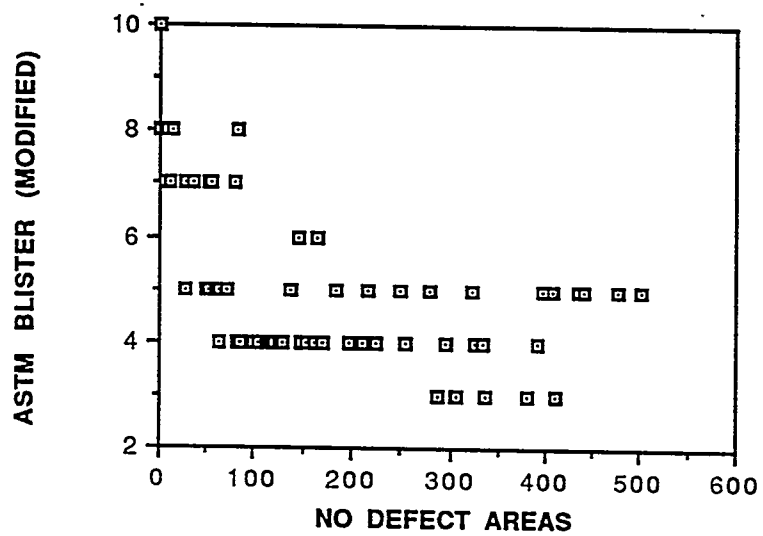


Figure 34 Comparison of ASTM vs. grid blister ratings, epoxy, 80 °C

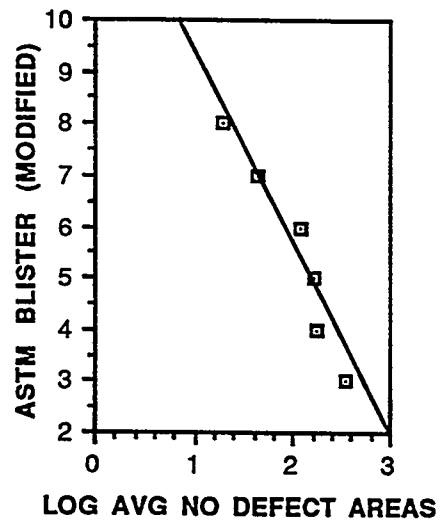


Figure 35 Linear correlation of ASTM vs. log of average grid blister ratings, epoxy, 80 °C

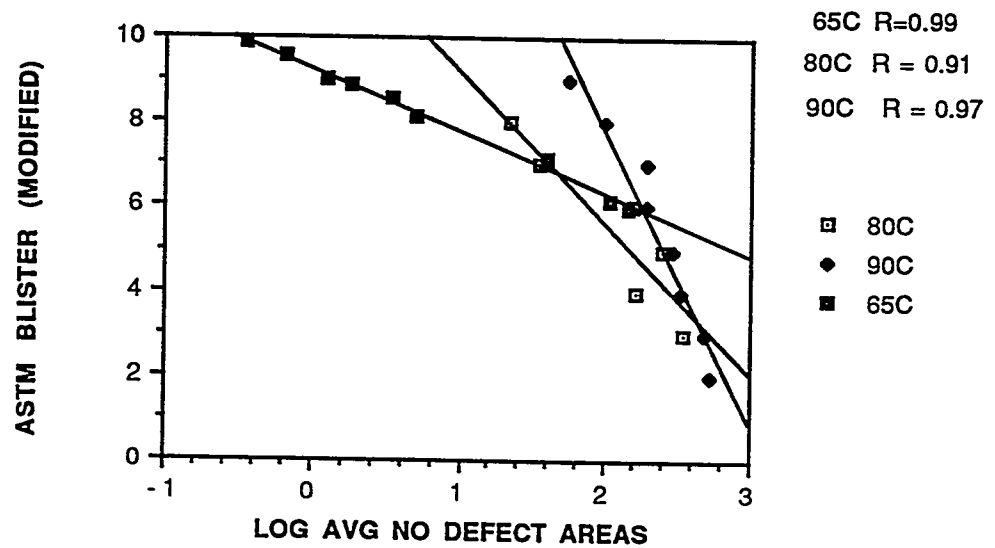


Figure 36 Linear correlation of ASTM vs. log of average grid blister ratings, epoxy, three temperatures

VI. RECOMMENDATIONS

1. TEST PROCEDURES

High-temperature immersion

High temperature can result in accelerated degradation in immersion conditions. However, the elevated temperatures can change the mode or mechanism of degradation. Before using elevated temperatures, it is recommended to first determine how the temperature will affect the physical properties of the coating such as flexibility and softening. For epoxy coatings, caution is advised for any temperatures above 75 °C.

Deionized vs. Salt Water

Deionized water at 90 ° has proven to be a more aggressive medium than tap water for promoting blistering, as expected. Tap water resulted in a greater degree of rusting. Salt water is expected to be even more aggressive toward rusting. Rusting is often related to defects in the coating. Thus, immersion in deionized water is recommended for testing the basic properties of the mating material. Other tests can then be used to determine the capabilities of applying a uniform film of suitable thickness.

High-Pressure Immersion Testing

Increased hydrostatic pressure also results in increased degradation of coated specimens. The overall effect of pressure is much more moderate than that of temperature. Thus, the increase in the pressure will not cause a dramatic reduction in the time for degradation. Based on the experiment on epoxy polyamides, it is recommended that for pressurized immersion testing, pressures in the range of 30 to 50 psig are suitable.

• Condensation Cabinet

The condensation cabinet can be operated as a multi-temperature chamber, but some precautions are needed. The degradation rate of coatings increases as the temperature is increased. For coatings with relatively low T_g s, like alkyds, the system may behave differently at the higher temperature, thus it is also important to examine the film properties at different temperatures. Another consideration is that at the higher temperatures, the rate of condensation on the coated surface may be reduced due to reductions of the difference in temperature between the panel surface and the water vapor. For experiments with a conventional condensation cabinet, a maximum temperature of 60°C is recommended for most systems.

The Cleveland condensing cabinet does not simulate the conditions of immersion. This was demonstrated in the case of the epoxy polyamide which showed extensive rusting in the Cleveland condensation cabinet, while in immersion in deionized water, the major degradation was by blistering.

• Acid Fog Salt Spray Cabinet

The use of acetic acid in salt fog does have an apparent accelerating effect on consuming zinc. This is attributed to the reaction of acid with zinc on the surface. However, the pattern of rusting formed may be different from that observed in conventional salt spray and in exterior exposure. In addition, it has proven difficult to maintain constant spray and collection conditions in the cabinet in the presence of acetic acid. The above deficiencies should be addressed for any future experiments utilizing acetic acid salt spray.

The salt spray test itself has been demonstrated to have severe limitations as an indication of outdoor service performance. The acetic acid salt spray will also suffer from those disadvantages.

- **Exterior Exposure**

For coatings designed for exterior exposure, testing conducted on outdoor racks is still considered the most reliable evaluation method. Data from Kure Beach and an industrial site demonstrate that using good statistical methods and sufficient replicates can yield relatively early data on mating performance. This corroborates recent work compiled by SSPC in the PACE (Performance of Alternate Coatings in the Environment) study. Thus, any accelerated testing program for exterior exposure should include a series placed in an aggressive outdoor site.

2. STATISTICAL METHODS

- **Time to Failure vs. Rating Over Time**

In comparing the performance of two or more coatings or coatings under different conditions, the concept of time to failure is an extremely useful evaluation tool. [t can supplement the usual, more conventional method of computing average ratings at a given time. In many instances the time to failure provides a more meaningful correlation to the field applications. For example, one is more likely to examine a tank or other structure for the percentage of the surface that has failed, rather than judge the need for maintenance or repair based on the "average" condition of the surface.

- **Failure Distributions**

Failure times and ratings of nominally identical coated specimens tend to vary, similar to behavior observed for other engineering materials. The manner in which the failures are distributed (i.e., narrow or broad range, shape of curve) can provide important information about the coating system sensitivity to variables like dry film thickness, cure conditions, etc. Failure distributions also provide a more sensitive means to differentiate two systems than conventional comparative methods.

- **Use of Multiple Replicates**

Because of the high variability in coating system performance, it is important to select an appropriate number of replicates for performance evaluation. This experiment has shown that a minimum of 10 to 15 replicates is required to construct distribution curves. To compute reliable average ratings, a smaller number of replicates may be acceptable. The usual practice of exposing two or three replicates is not a statistically sound approach. A practical compromise to ensure representative average ratings may be to use 5 or 6 replicates.

3. **EVALUATION METHODS**

The quantitative visual evaluation (grid) method has proven to be very useful and practical. This technique requires additional rating time but yields additional information which is extremely valuable. It allows more precise comparison of performance among two or more systems. It also allows one to compute more precise curves depicting performance versus time.

The grid method is recommended as a supplement to conventional ASTM methods. The use of the latter is still suggested, because of researchers' familiarity with those methods, and to establish better degrees of correlation between the two rating schemes. The grid method is especially suitable for evaluating rusting. Particular care is required when using the method to evaluate blistering to ensure consistency among raters.

APPENDIX A

MAJOR AREAS OF SHIPS AND TYPES OF COATINGS USED

Steel ships exposed to the ravages of a sea-salt atmosphere require special coatings to preserve the hull and to promote a "ship-shape" appearance. Surfaces below the water, above the water, exterior and interior all require special coating design considerations. A review of the different environments involved and the coatings used to combat these environments is described below.

Underwater Surfaces: The underwater area is often referred to as Keel to Light Load Line. The underwater steel is immersed at all times and is subject to continuous galvanic corrosion and fouling. Within harbors and inland waterways, the flat bottom and bilge areas suffer from damage by occasional scraping and grounding. If idle for more than a few days, fouling attaches and causes drag on the hull, which increases fuel costs.

This part of the vessel requires an anti-corrosive (A/C) coating which serves as a barrier between the steel and the underwater elements. The A/C coating is then topcoated with an anti-fouling (A/F) coating. The A/C should be abrasion resistant and water impermeable. Most owners use two coats of coal tar epoxy or pure epoxy at 4-5 mils dry film thickness (DFT) per coat. The A/F systems commonly used are of two general types. One is a "conventional", copper-oxide, vinyl type at two to three mils at 2 mils DIT per coat. The other is an "ablative" type, which has organo-tin type pigments in a polishing co-polymer binder. This coating is usually applied in three coats at 3 mils DFT per coat. This latter type wears away at a controlled rate. The wearing mechanism results in a smooth surface which saves fuel and provides an unsound footing for hard shell organisms. Of course the A/C and A/F coatings have to be compatible with each other. Each coating manufacturer will have a "system" or "systems" designed for the trade patterns and service life that an owner requires. Most vessels today are equipped with external cathodic protection to supplement the coatings.

Boottop. This area is between the Light Load Line and the Deep Load Line. Light Load is a condition when the vessel is not fully loaded or in ballast. Deep load is when the vessel is loaded to capacity. This zone is subject to immersion under Deep Load Conditions and wind and water in Light conditions. It is exposed to splash and ultraviolet light when out of water and to immersion same as the underwater surfaces when in the Deep Load condition. When maneuvered by tugboats and alongside piers and docks, this area is scuffed and rubbed, causing mechanical damages to the coating.

A heavy-duty coating system would include an inorganic primer, an epoxy midcoat, and a suitable finish coat. A typical system would be first coat inorganic zinc, 3 mils DFT, 2nd coat high-build epoxy at 3-5 mils DFT, and 3rd coat a polyurethane or epoxy at 2 mils DFT. This system can also be used without the inorganic zinc primer; substituting an epoxy primer at 2-4 mils DFT.

Topsides: This is the area which is above the water most of the time. It is called Deep Load Line to Rail. It is here that the color and gloss are important for aesthetic reasons. The steel must be protected from salt spray and occasional immersion in heavy weather, and the coating must resist in addition the ravages of ultraviolet light and temperature changes from day to night.

This area may require less coating DFT than the Boottop because it has less abrasion from dockside bruises and it is not subject to long periods of continuous immersion. Usually, however, the mating system is the same as the Boottop area.

Decks: The Main deck is subjected to abrasion from traffic and loading operations as well as salt spray, green seas in bad weather, and ultraviolet and temperature changes. The other weather decks are usually not subject to abrasion from work loads.

The coating system varies depending upon the owner's preference. If the ship's crews employ regular maintenance, a 4-coat chlorinated rubber or alkyd system at 2 roils DFT per mat or a 2-mat epoxy with modified vinyl, epoxy, or acrylic is often used. For a maintenance-free deck, an inorganic zinc, permanent primer at 3 roils DFT is used under an epoxy midcoat with 1 or 2 mats of finish that is compatible with maintenance paints. The latter is necessary for occasions when the owner wants to dress up the deck for appearance between drydock repair periods.

Typical systems in addition to the one above are A) 1 coat of inorganic zinc at 3 roils DFT plus 1 coat of epoxy at 2-4 roils DFT, and 2 mats of modified vinyl or acrylic at 1.5 to 2 roils per coat, or B) 1 mat of epoxy primer at 2-4 roils DFT in lieu of the inorganic zinc and all other coats the same. The 4-coat chlorinated rubber or alkyd maintenance system requires continuous maintenance by the ship's crew. For major work areas, a non-skid additive is added to the final coat.

Superstructure: The superstructure is the part of the vessel above the main deck level. [It includes deck hoses, forecastle, pump rooms, and the engine room and accommodation houses. Unlike the decks, it is difficult to maintain by the ship's crew. These areas stand out above all the rest and aesthetics are important. Color and gloss finishes are more important here than in other areas (except Topsides).

Any of the Boottop or Topsides coating systems can be used here. It is important not to have rust streaking down so inorganic zinc, permanent primer, is often used.

General Interior Hull: Owners have a choice of many different kinds of coating systems including vinyl, chlorinated rubber, epoxy, urethane, alkyd, and modified types of vinyl, epoxy, and acrylics. The choice depends upon 1) the trade the vessel is in, 2) the geographic location and climatic conditions where the mating is to be applied, and 3) the economics. Life-cycle economics will usually show that a heavy-duty system pays for itself in the long term. But many owners opt to use cheaper paints and recoat frequently, i.e., between regular repair periods, or simply let them go if the aesthetics are not a consideration. Each generic type has its advantages and disadvantages. The alkyds are least resistant to the elements and need constant touch-up maintenance. The vinyls and chlorinated rubbers are chemically resistant but are thin and require more coats than the higher build products. They are also soft and are damaged easily from impact and abrasion. The epoxies are durable and abrasion resistant but they have poor color retention and gloss. They also have intercoat adhesion problems and do not cure at cold temperatures. The urethanes have good color and gloss, are suitable for cold temperature applications, but are expensive on a price per gallon basis. All of these factors must be weighed in the selection process.

Fuel Tanks: Tanks that carry fuels and petroleum products are coated for two reasons. One is to protect the steel surface from corrosion; the other to protect the fuel or cargo from being contaminated with rust. The coating must be compatible with the oil product and be able to withstand sea atmosphere, washing, slops, etc. when the tank is empty or partially full.

The coating system is usually a 2- or 3-coat epoxy at 2-4 mils DFT per coat for most fuels. Other generic types such as phenolics and epoxy phenolics are sometimes used.

Ballast Tanks: These tanks are ballasted with sea water under Light Load conditions and are empty during loaded conditions. Some vessels use ballast tanks only for heavy weather, and others may carry ballast partially full as needed to maintain proper draft. This continuous and intermittent exposure to sea water is similar to the exterior Boottop and underwater areas. However, the Boottop and bottom hull can be repaired periodically during drydock repair periods. The interior tanks require extensive staging, lighting, ventilation, dehumidification, grit removal, and other expensive preparation and application in which case it is prudent to apply a "permanent" coating system at new construction. Such a coating system will last 10 years with little or no maintenance.

Typical coating systems are 1 coat of heavy-duty inorganic zinc at 3-5 mils DFT, or 2 coats of coal tar epoxy or pure epoxy at 4-6 mils DFT per coat. Also, it is recommended that back-up sacrificial anodes be installed and maintained to prevent edges, welds, and minor defects from breaking down during the service life of the coating.

Special Areas: Hot surfaces, such as steam lines on deck, require special coatings. Potable water tanks require coatings approval by the U.S. Public Health Service. Feed water tanks, voids, cofferdams, peak tanks are all examples of special areas which require different coating systems. Each must be designed to suit its special needs.

Preconstruction Primer (PCP): For new construction, a PCP is usually used. Sometimes the PCP is removed and the first coat of the coating system is applied over the freshly prepared, bare steel surface. Other builders may elect to use the PCP as the first coat of the system. In such case the PCP has to be compatible with the service intended and the coating system to be applied over it. Each paint manufacturer has a PCP designed to go with his particular coating system. However, sometimes one PCP is used under several different other vendors products. This is a special subject which has to be addressed early in the specification design stage.

Typical PCPS are epoxy zinc-rich, inorganic zinc, or zinc-free epoxy primers applied at 0.75-1.25 mils DFT.

**GENERAL PAINTING AND COATING SPECIFICATIONS
For Exterior Hull Surfaces and Interior Tanks***

Line	Location	1st Coat	2nd Coat	3rd Coat	4th/5th Coat	Minimum Total DFT
1.	UNDERWATER HULL (Keel-LLL)	Coal Tar Epoxy ¹ 4-6 mils	Coal Tar Epoxy ¹ 4-6 mils	Vinyl, Copper Oxide, A/F 1.5-2 mils	Vinyl, Copper Oxide, A/F 1,2 1.5-2 mils	12.5 mils
2.	Alternate I (Note 4)	Epoxy ¹ 4-6 mils	Epoxy ¹ 4-6 mils	Ablative-Type A/F 1,3 3-4 mils	Ablative Type A/F 1,2,3 3-4 mils	17 mils
3.	Alternate II (Note 4)	Epoxy ¹ 4-6 mils	Epoxy ¹ 4-6 mils	Vinyl, Organo-Tin Copper Oxide, A/F 1.5-2 mils	Vinyl, Organo-Tin/ Copper Oxide, A/F ² 1.5-2 mils	12.5 mils
4.	BOOTTOP (LLL-DLL)	Inorganic Zinc Silicate 2-3 mils	Epoxy ^{1, 5} 3-5 mils	Polyurethane 2-2.5 mils		7 mils
5.	Alternate I	Inorganic Zinc Silicate 2-3 mils	Epoxy ^{1,5} 3-5 mils	Epoxy 2-4 mils		7 mils
6.	Alternate II	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	Epoxy 2-4 mils		6 mils

* Near-White metal (SSPC-SP 10) is minimum recommended surface preparation for all coating systems presented.

¹ Apply topcoats within manufacturer's specified time intervals.

² Minimum drying time as specified by manufacturer shall be allowed between last coat and undocking of ship.

³ Dry film thickness shall be adjusted for intended service life, i.e., between drydockings.

⁴ Any of the antifouling paints can be used over the selected anticorrosive system.

⁵ This product is to be specifically formulated for topcoating inorganic zinc. Otherwise, a tie-coat, 1-1.5 mils DFT should be applied here as an extra coat.

LLL = Light Load Line DLL = Deep Load Line

A/F = Antifouling DFT = Dry Film Thickness

**GENERAL PAINTING AND COATING SPECIFICATIONS
For Exterior Hull Surfaces and Interior Tanks**

Line	Location	1st Coat	2nd Coat	3rd Coat	4th/5th Coat	Minimum Total DFT
7.	TOPSIDES	Inorganic Zinc Silicate 2-3 mils	Epoxy ^{1,5} 2-4 mils	Polyurethane 2-2.5 mils		6 mils
8.	Alternate I	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	Epoxy 2-4 mils		6 mils
9.	Alternate II	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	Mod. Epoxy or Mod. Vinyl or Mod. Acrylic 1.5-2 mils	Mod. Epoxy or Mod. Vinyl or Mod. Acrylic 1.5-2 mils	7 mils
10.	DECKS	Inorganic Zinc Silicate 2-3 mils	Epoxy ^{1,5} 2-4 mils	Mod. Epoxy or Mod. Vinyl or Mod. Acrylic ¹ 1.5-2 mils	Mod. Epoxy or Mod. Vinyl or Mod. Acrylic ¹ 1.5-2 mils	7 mils
11.	Alternate I	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	Mod. Epoxy or Mod. Vinyl or Mod. Acrylic 1.5-2 mils		5.5 mils
12.	Alternate II	Chlor. Rub. Primer ¹ 1.5 - 2 mils	Chlor. Rubber Primer ¹ 1.5-2 mils	Chlor Rubber Finish ¹ 1.5-2 mils	Chlor. Rubber Finish 1.5-2 mils	6 mils
13.	Alternate III	Marine Alkyd Primer ¹ 1.5-2 mils	Marine Alkyd Primer ¹ 1.5-2 mils	Marine Alkyd Finish ¹ 1.5-2 mils	Marine Alkyd Finish 1.5-2 mils	6 mils

**GENERAL PAINTING AND COATING SPECIFICATIONS
For Exterior Hull Surfaces and Interior Tanks**

Line	Location	1st Coat	2nd Coat	3rd Coat	4th/5th Coat	Minimum Total DFT
14.	SUPERSTRUCTURE	Inorganic Zinc Silicate 2-3 mils	Epoxy ^{1,5} 2-4 mils	Polyurethane 1.5-2 mils		5.5 mils
15.	Alternate I	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	¹ Mod. Epoxy or Mod. Vinyl or Mod. Acrylic ¹ 1.5-2 mils		5.5 mils
16.	Alternate II	Chlor. Rubber Primer ¹ 1.5-2 mils	Chlor. Rubber Primer ¹ 1.5-2 mils	Chlor. Rubber Finish ¹ 1.5-2 mils	Chlor. Rubber Finish 1.5-2 mils	6 mils
17.	Alternate III	Marine Alkyd Primer ¹ 1.5-2 mils	Marine Alkyd Primer ¹ 1.5-2 mils	Marine Alkyd Finish ¹ 1.5-2 mils	Marine Alkyd Finish 1.5-2 mils	6 mils
18.	SEGREGATED BALLAST TANKS	Inorganic Zinc Silicate 3-5 mils				3 mils
19.	Alternate I	Coal Tar Epoxy ¹ 4-6 mils	Coal Tar Epoxy 4-6 mils			8 mils
20.	Alternate II	Epoxy ¹ 4-6 mils	Epoxy 4-6 mils			8 mils
21.	Alternate III	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	Epoxy 2-4 mils		6 mils
22.	FUEL TANKS	Epoxy ¹ 4-6 mils	Epoxy 4-6 mils			8 mils
23.	Alternate I	Epoxy ¹ 2-4 mils	Epoxy ¹ 2-4 mils	Epoxy 2-4 mils		6 mils

APPENDIX B

OVERVIEW OF ACCELERATED TESTING PROCEDURES

Extracted from FSCT Report
"Survey of Accelerated Test Methods for Anti-Corrosive Coatings"

There are many internal stresses acting on a coating to dislodge it from the surface or to cause internal cracks, voids, or separations. (Croll, 1978) To this are added various external stresses produced by the interaction with the environment and substrate. In this section, the discussions will be about the external stresses, which are those over which the experimenter has the greatest degree of control.

Exposures can be classified into the following categories:

- artificial accelerated
- natural atmospheric (unaccelerated)
- accelerated natural

A non-artificial (i.e., "natural") exposure is one which occurs either in nature (e.g., outdoor locations such as test fence) or in service (e.g., interior or exterior of process vessel). An "accelerated" exposure is one in which the stress factors are higher than would occur under normal service conditions. Most laboratory test chambers are artificial accelerated tests (e.g., salt spray), whereas the real-time, or service tests (e.g., Florida test station) are unaccelerated natural exposures. (Garlock and Sward, 1972) Accelerated natural tests are those in which an extra stress (e.g., water spray or concentrated sunlight) is placed on a coating exposed in natural (i.e., atmospheric) conditions.

A. ARTIFICIAL ACCELERATED EXPOSURES

This is the type that has received the most attention and also the most criticism. The basic idea is to simulate or reproduce the stresses or conditions of real-world exposure but at a more intense level. It is clear that laboratories cannot produce all the stresses inherent in an outdoor environment. It then becomes necessary to identify the most significant stresses.

1. Types of Stresses

Artificial accelerated exposures can be classified based on the types of stress and the manner in which they are imposed. One of the most important distinctions among exposure tests is whether they are constant stress or cyclic stress.

Constant Stress Tests

In "constant stress" chambers, a stress (or a combination of stresses) is applied monotonically for an extended period of time. Examples of such tests are salt spray, temperature/humidity, condensing humidity, and immersion cabinets.

A temperature/humidity cabinet is an example of a constant stress chamber, which can provide variations of a standard atmosphere. A standard “unpolluted” atmosphere consists of atmospheric gases: nitrogen, oxygen, carbon dioxide and water vapor. Of these only oxygen and water vapor are expected to influence the performance of coated metals. For oxygen, the concentration is relatively constant, but the concentration of water vapor (i.e., relative humidity) varies considerably with location, time of day, and season.

The effect of the water vapor concentration can be studied by varying the relative humidity at a given temperature. It is also of interest to observe the coating's performance at various temperatures under constant humidity. Martin and McKnight (1985) studied the reactions of acrylic and alkyd matings exposed to a humidity chamber at constant relative humidity (at several temperatures). By excluding other stresses, the researchers were able to show an Arrhenius plot of degree of degradation vs. inverse temperature, and estimate activation energies. In another major investigation using a constant single stress test, the SSPC has immersed epoxy panels in pure (deionized) water at varying temperatures. (Appleman, 1988)

Most constant stress chambers, however, use multiple stresses in order to degrade the coating. Immersion in salt water, for example, exposes the coating to the actions of both water and salt. Similarly, the salt spray test (i.e., ASTM B-1 17) subjects the coating to a mist of salt particles and water.

In addition, many of these tests are run at elevated temperatures. Temperature is one of the most crucial variables in any type of artificial exposure. The temperature normally increases the rate of most chemical processes, including corrosion and film degradation reactions.

The temperature has a major effect on the rheology of the cured coating. Most coatings are formulated to maintain an integral film over a range of temperatures; however, if that range is exceeded, a mating could undergo reactions or transformations that would not occur under natural conditions. An important concept here is the glass transition temperature (T_g), the ramifications of which have been thoroughly described. (Hill, 1987)

A major criticism of tests such as the salt spray is that they do not allow relaxation of the stress, such as a drying-out period. Because the level of stress normally changes periodically under “natural” renditions, constant stress chambers may produce degradation that is not observed in nature. Reviews of accelerated tests are given by Funke (1979), Lee and Money (1984), Lindberg (1975), Reeve (1986) and Wicks (1987).

2 cyclic stress Tests

Cyclic Salt Spray

Cyclic accelerated tests provide a regular variation of stress. In some cases, the coating is simply removed from the stress (e.g., removing a coated panel from an immersion bath for a few hours). The Kesternich test is another such example. (Berke and Townsend, 1988) The

specimen is exposed to a combination of water vapor and sulfur dioxide for 8 hours, followed by 16 hours in which the chamber is open to the ambient laboratory environment.

Some cyclic tests provide alternating stresses rather than merely relaxing a stress. The most common examples of these are the various so-called “accelerated weathering” chambers, such as the Weatherometer. The cycle here is a xenon or carbon-arc lamp (to simulate the sunlight spectrum) followed by a water spray (to simulate rain).

Recently many researchers and equipment manufacturers have come to believe that improvements in accelerated testing lie in developing better and more representative cyclic tests. This development of new testing procedures has proceeded along two distinct directions. One group has emphasized development of cyclic variations of salt spray and other tests designed to simulate corrosive environments; the other trend has focused on the modification and refinement of accelerated weathering devices where the concern is for appearance properties (primarily gloss).

A cyclic Tests for Corrosion

Cyclic Salt Spray

The most widely used accelerated test for corrosion testing is the salt spray test. This is true in spite of the vast amount of unfavorable comments that have been heaped on this test for many years. (Hare, 1982; Liu, 1981) A number of researchers have attempted to reduce the disadvantages of the salt spray by using it in conjunction with other accelerated tests, thus the highway agencies and automobile manufacturers have developed their own specialized cyclic corrosion tests. (Assoc. of Auto Ind. 1986; Tinklenberg, 1986) in these tests, the specimens are rotated among 2 or 3 separate chambers. In this manner the coatings receive a relaxation period for each of the stresses, as well as receiving a variety of stresses. Most of these laboratories are convinced that these cyclic tests provide a more realistic simulation of the field exposures for bridges, automobiles or other structures. Several of these cycles have recently been reviewed.(Anonymous, 1980; Appleman, 1982 and 1989; Hare 1982; Jussiaume, 1984; Standish, 1985)

A more sophisticated approach toward cyclic corrosion testing is the development and use of specialized test chambers which automatically produce the desired cycle. An early example is the “Prohesion” cabinet developed by Timmins (1974) The test was dubbed “Prohesion” based on the slogan “Protection by Adhesion”. The researchers at British Rail thought that this test was better able to determine the ability of a coating to adhere under adverse conditions, a property they felt to be critical to performance. This test was subsequently transferred to Mebon Paint Company, which developed a standard cabinet which included the forced circulation of warm dry air to dry off the specimen surfaces. This is an important feature which distinguishes Prohesion from other cabinets in which the salt cycle is turned off, but does not necessarily provide a mechanism or opportunity for the specimen to dry off prior to the next salt cycle. (Anonymous, Pigment & Resin Technology, 1980) Typically, the Prohesion cabinet differs from the conventional U.S. salt spray cabinet in that the salt used is a mixture of ammonium sulfate and sodium chloride (0.5%). The later is much lower than the 57. stipulated in ASTM D-117. The British authors claim that the use of the lower salt along with ammonium

sulfate (the "Harrison's solution") provided a better simulation of atmospheric corrosion effects.

There are several other suppliers of cyclic salt spray cabinets capable of automatic cycling. These include the units from Erichsen, H. Kohler, Liebisch, and Hotpack. Each of these includes microprocessor controls and variable cycles of the salt spray. These may require some modification to provide circulation of drying air. Two major U.S. manufacturers, Singleton and Englehard/Harshaw, have indicated that they can produce a salt spray cabinet with automatic cycling, but that such units are not currently among their standard options and would have to be custom built. Among the cabinets described, there are a range of sizes and temperature ranges available. One is now commercially sold in the UK and the manufacturer and several users have claimed its superiority over conventional salt spray. In particular, Skerry (1987) has presented data suggesting that the crystal structure and appearance of corrosion products produced in outdoor exposures is much more closely simulated by the Prohesion cabinet than by salt spray. Other cyclic corrosion tests have also been evaluated (Nowak et al., 1982, Saatweber, 1983).

Cyclic Immersion Testing

There has been relatively little commercial development of cyclic cabinets capable of immersion. The most innovative one identified was the Envirotest. (Tator, 1989) In this apparatus, the panels are attached to a rack which rotates through a 360 degree arc. At the bottom of the arc, the panels are immersed in any of a number of solutions, but most commonly salt water or fresh water. The top of the cabinet is fitted with ultraviolet or heat lamps which subject the specimens to heat and ultraviolet light, thereby causing the surfaces to dry out. The duration of the cycle can be varied as well as the other parameters of the chamber. The manufacturer has noted that it may be possible to introduce gases during the intermediate portions of the cycle through ports. This feature has not yet been made available. Presently, there are about three or four such units being used.

A cyclic immersion test is also described in NACE TM-01-84. In this method, the specimens are placed in an immersion tank which, through some various siphons and pumps, is periodically evaluated, allowing the panels to dry out. Variations of this type of apparatus have also been constructed in the SSPC and other laboratories. All that is required to construct a cyclic immersion test is to add a siphon with a timer to conventional immersion tanks.

Cyclic (Non-Condensing) Humidity

Non-condensing humidity refers to conditions of high humidity, but less than 100% relative humidity. There are a number of humidity cabinets which can provide humidity ranges as low as 20% and as high as 980%. Because of the close relationship with temperature, most humidity cabinets also have the capability of varying temperature. The relative humidity and temperature ranges are shown in Appendix B of the full FSCT report (Appleman, 1990) for some commercially available equipment. There are numerous chambers capable of automatically cycling between one or more temperature/humidity conditions. These conditions, however, by themselves, are not very aggressive to most coatings, except at very high temperatures which cause breakdown of the polymers. Thus, these cabinets have not been

widely used for accelerated degradation, although they would be suitable for studying subtle changes in properties as a function of temperature and humidity variations.

Cyclic Condensing Humidity

In order for condensation to occur uniformly and consistently, the temperature of the specimens should be several degrees lower than the temperature of the 100% water saturated vapor. This condition is achieved in the Cleveland Humidity Cabinet, which is described in ASTM D 4585 and ASTM D 2247. Condensing humidity can be cycled in several manners. (Grossman, 1972) Turning off the heating element periodically is not a very effective way of cycling "off" the condensation because of the time required for the specimens to cool down to ambient temperature and dry off. One approach used years ago by developers of the cabinet is to introduce a fan into the chamber to air dry the panel. (This is described in Sections 5.6 and 7.5 and ASTM D 4585). The introduction of UV lamps into the cabinet (see later description) also results in effectively drying out the surface.

Cyclic Sulfur Dioxide

Although several corrosive gases are sometimes used in accelerated testing of paints, sulfur dioxide is by far the most important for evaluating the corrosion resistance of mating systems and metals.

One of the oldest and most widely known sulfur dioxide corrosion tests is the Kesternich test. In this test, both sulfur dioxide and water vapor (and sometimes carbon monoxide) are introduced into a chamber with suspended metallic or coated specimens. These specimens are exposed to the gases for a fixed period, usually eight hours (corresponding to a normal work day). (Haagen, 1983) More recent developments in gas testing chambers include microprocessor controls which automatically vent cabinets to the ambient conditions. Typically, the cycles may be run for 7, 14, 30, or more days. In some versions of the test, the specimens are exposed for longer periods of time (close to 24 hours) with a very short dry-out period. The ASTM has developed its own version of this test, "Standard Practice for Conducting Moist SO₂ Test (ASTM G 87; Berke and Townsend, 1988).

SCAB Corrosion Test

Automobile manufacturers have developed a cyclic corrosion test known generically as SCAB test. (Wyvil, 1982; Miyoshi et al, 1982) The name refers to the appearance of small rust filled blisters at areas of paint breakdown. A SCAB test developed by Ford (referred to as the APGE [Arizona Proving Ground Equivalent]) is as follows: scribed panel immersed or sprayed with 5% salt for 15 minutes; panel allowed to drain and dry for one hour and 15 minutes; panel then placed in conditions of high temperature (120°F and high humidity (85-90%)) for 22-1/2 hours. This procedure is repeated daily. The test is typically run for 10 to 20 weeks and is claimed to be effective in reproducing the type of corrosion obtained by the exterior APGE test. (Smith, 1988) That test is performed on a vehicle which is driven a number of times through a salt laden gravel road and a salt spray facility and is then placed in a high temperature and humidity chamber for eight hours and dried out for eight hours. After each 10 cycles (days) the vehicles are run through an automatic car wash and are inspected. A number

of versions of the field exposure test have also been developed. Presently there is a major cooperative effort being undertaken among the Society of Automotive Engineers (SAE), American Iron and Steel Institute (AISI), ASTM, and automotive manufacturers and coating suppliers to establish an industry-wide standard, cyclic corrosion test for automotive coatings.

3. Cyclic Tests for Accelerated Weathering

One of the most important causes of coating degradation is ultraviolet light from the sun. There are several sources of artificial UV light, including fluorescent bulbs, and xenon and carbon-arc lamps.

Weatherometers

The earliest type of weathering chamber developed is the weatherometer, which used carbon or xenon source lamps with and without filters. Early experiments had shown that by including alternating periods of moisture, the degradation was greatly accelerated. These cycles allowed a hot/cold as well as a wet/dry cycle. (Kinmonth and Norton, 1977) Because the UV light also heats up the surface, the specimens cooled during the dark portion of the cycle, causing a thermal cycling (fatigue). In addition, any water absorbed during the moisture cycle would tend to be evaporated during the light cycle, causing an absorption/desorption cycle stress. (Birchenough, 1986; Boxall, 1984; Kuenstler and Shur, 1968; Reeve, 1986)

Over the years, research groups and manufacturers have developed and evaluated a large number of different designs, cycles, and controls. (De Tommaso et al, 1980; Hearst, 1970; Helmen and Hess, 1979; Stieg, 1966) Among these are the following:

- Twin-Carbon Arc
- Xenon Arc
- Dew Cycle Weatherometer

The closest simulation to sunlight is achieved using xenon lamps with suitable filters. (Hoffman and Vogel, 1985)

A special task group of the National Coil Coaters Association (NCCA) evaluated 47 coatings in five accelerated chambers and three South Florida sites for 18 months. The group found some evidence of correlation, but because of the high standard error, the data had poor predictive value. The overall conclusion was that for 18 months exposure there is no strong correlation between any of the accelerated weathering devices and Florida exposure. They recommended that for future work, each generic coating be tested in a separate program, that panels be exposed for five years, and that variables such as color, gloss, and exposure angle be minimized. (NCCA, 1978)

In addition, a number of comprehensive, detailed statistical evaluations have been conducted to determine how well these methods correlate with one another and with outdoor exposure sites. (Blakey, 1985, Ellinger 1977, Hoey, 1974; Hoffman and Vogel, 1985; Goering et al, 1981, Kamal and Saxon, 1967; Lindberg, 1975; Scott, 1980)

Fluorescent UV Tests

The cycles incorporating fluorescent UV light were developed by Q-Panel Company, and modelled after the Cleveland Condensing Cabinet. The UV cycle was provided by a series of 8 fluorescent lamps. During the dark cycle, a tray of water was heated to produce water vapor which would condense on the mated test specimen, the outside of which was at ambient temperature. Typical cycle used was 4 hours UV light and 4 hours condensation, the method was standardized as ASTM G-53. Other manufacturers also designed equipment to meet this standard. (Grossman, 1977; Brennan, 1982)

The major criticism directed at this technique is that the UV-B lamps (most commonly used) are not suitable simulations of the solar spectrum. In particular, the UV-B lamps emit radiation of higher energy (lower wavelength) than sunlight; this high-energy radiation is absorbed by certain polymers (e.g., urethanes) which are thereby degraded by reactions that would not have occurred under natural sunlight. (Mar and Schirmann, 1987)

In response to this limitation, a major manufacturer of such cabinets has recently introduced fluorescent bulbs of lower energy, the UV-A lamps. Because these bulbs have lower intensity than UV-B lamps, at the highest energy solar radiation bands, they require a substantially larger time to produce a given level of degradation. (Brennan, 1987)

The standard UV-B lamps have nevertheless gained an extremely wide acceptance in the industry for several reasons. The apparatus is simple and inexpensive to operate and maintain. Degradation of coatings as measured by loss of gloss or color is rapid. Many users employ the test primarily as a screening test or quality control test. It provides relative information on the UV and moisture resistance of a coating.

Many of the most sophisticated users are convinced that no weathering cycle can ever accurately simulate solar radiation, their use of fluorescent UV lamps is tempered by an awareness of their limitations for general predictions and the unnatural effects of the highest energy portion of the spectrum. These organizations will normally also conduct outdoor field trials in parallel with or as a follow-up to all accelerated laboratory tests to corroborate preliminary findings. (Fisher, 1984)

Probably, however, most users of the fluorescent UV test are not fully aware of these limitations, in spite of good technical literature furnished by the manufacturers. These users risk erroneous conclusions regarding relative UV resistance and suitability of certain coatings or polymers for various applications.

An important conclusion is that the correlations obtained are very specific to the type of polymer or coating system studied, and to the method. (Simms, 1987)

B. NATURAL ATMOSPHERIC EXPOSURE TESTING

The vast majority of industrial maintenance coatings are exposed under atmospheric conditions. "Real-Time" atmospheric exposures are considered the most reliable by researchers.

in the industry, as the coating is tested in the type of environment in which it will ultimately be exposed; there are no "artificial" conditions imposed on the coated substrate. The coating is exposed simultaneously to all the potential stress factors, e.g., sunlight (ultraviolet radiation), rain, dew, atmospheric pollutants (acid rain, ozone), and temperature and humidity fluctuations. There are, however, some major drawbacks to the use of outdoor exposure tests, some of which are quite obvious. These include the following:

- Variability of "natural" environment.
- Multiple stresses
- Lack of controls
- Slow degradation

1. Variability of Environment

Atmospheric exposures are inherently variable. First of all, there are enormous variations among different parts of the country, (i.e., Florida vs. California vs. the Northeast). A long-term average of temperature or solar radiation or relative humidity would show enormous differences among these locations. Even within a smaller geographical area, the proximity of industry or local variations in weather patterns would also result in significant differences in the exposure environment. Furthermore, studies have shown that significant fluctuations will occur even at a single test site from year to year or season to season. This complicated the efforts to establish correlations between accelerated tests and South Florida exposures, which has been accepted as the industry standard "natural" site.

It should be quite obvious that there is no single "standard" outdoor exposure site. One approach for utilizing outdoor sites is to pick one or more sites representative of the actual exposure conditions and to expose specimens for several years. By doing this type of testing with standard control coatings, one can establish performance parameters, including average durability and, most importantly, a degree of confidence and knowledge of expected fluctuations. (Scott, 1977, Scott, 1983)

2. Multiple Stresses

Because of the large number of stresses that are acting upon a coating in the atmospheric environment it is extremely difficult to isolate the effects of specific stresses. Thus, if a coating fails, it is difficult to determine what the most important factor was, (i.e., sunlight, condensation, thermal cycling, or a combination of these factors).

3. Lack of Controls

This factor relates directly to the variability of the exposure. For a test to be most useful, it should provide reproducible results with replicate specimens. As noted above, 2 identical panels exposed at the same location at different years or months will not be exposed to the same environment, and hence not give necessarily comparable performance. For planning a test program, a researcher needs to know that a standard mating will degrade in (say) 6 months, so that a candidate can be judged on its ability to last for a comparable period. This type of consistency in performance is difficult to achieve using outdoor exposure testing.

Slow Degradation

Finally, and perhaps most significantly, the outdoor degradation of most protective mating systems is extremely slow. With today's technology it is not uncommon for coating systems to last 10 to 20 years, even in aggressive environments. Waiting even 5 years is not a very practical approach to developing new products for the industry. (Bullett, 1968)

In some situations where conventional coatings only last 2 or 3 years, outdoor exposure testing in these same environments has proven beneficial. This approach may also apply for coatings applied over poorly prepared surfaces or under other adverse conditions with consequent expected lifetime of only a few years.

For the majority of applications, outdoor degradations are too slow for screening new products. A number of researchers have attempted to speed up the natural process by increasing the intensity of certain stress factors, while retaining the multiple stresses imposed by nature.

C. ACCELERATED NATURAL EXPOSURES

Some examples of these "accelerated natural" exposures are as follows:

- water or solution spray
- concentrated sunlight
- concentrated heat (black body temperature)
- periodic immersion
- specialty treatments

1. Outdoor Water or Solution Spray

Several agencies have established tests with periodic spraying of salt, water, or acidified water onto specimens exposed in outdoor panel racks. This treatment is designed to accelerate the degradation induced by a combination of salt, acid, and moisture, which are extremely corrosive to exposed steel and can also attack the coating itself. Spray frequency ranges from once a week to several times per day. The spraying may be automated or manual. "Acceleration factors" of up to tenfold have been attributed to this type of testing, but little confirmed data have been presented. (Crewdson and Lane, 1987; Lampe and Saarnack, 1986)

2. Concentrated Sunlight

This is a technique in which specialized mirrors (Fresnel reflectors) are used to reflect sunlight to the location where the panel is exposed. The mirrors are rotated to follow the sun, so that the maximum amount of UV radiation is received. This technique was commercialized by a testing laboratory based in Arizona. A similar unit has recently been developed by a Florida testing firm. A variation of the test uses periodic water spray to simulate the effect of rain.

Considerable evaluation and analysis of this technique has documented the increase in amount of solar radiation received. Several matings and equipment manufacturers and users in the industry have recommended using the total amount of solar radiation as a measure of the amount of exposure, rather than the length of time exposed. This concept is discussed more fully in the references cited. (Zerlaut and Ellinger, 1981; Morse, 1982; McEwen et al., 1987; Scott, 1982)

3. Black-Rodiation

In this technique, racks are instructed with heat absorbing material so that the panels will be heated to above the ambient temperature. The increased temperature will presumably increase the rate of any polymeric degradation or corrosion reactions because of the temperature dependence of most chemical processes. In this test, the panels do not receive increased levels of UV radiation. This test has been widely used by the automotive industry, which has developed several standard test methods. A recent study by ASTM Subcommittee D-127 (Accelerated Testing) has shown good correlation after 24 months of different South Florida locations of the 5 degree Black Box exposure (ASTM G7). (Morse, 1982)

4. Specialized Techniques

Periodic Immersion: A testing agency in Florida has developed a device which periodically cascades test panels with seawater. This is similar to the outdoor salt spray described above, except that the panels are subjected to a surge of water which immerses them for a short period and has some erosive or washing force.

Accelerated Antifouling Test: The rotating drum test has been used to evaluate coatings resistance to fouling. The normal test of a coating's marine fouling resistance is immersion of test panels in static sea water for periods of several months to several years. In the rotating drum method, the panels are rotated in seawater at a speed of several knots. This is considered to give better simulation of the service of an anti-fouling mating on a ship in motion. An ASTM test is also being developed for this procedure. Alternate approaches have been developed using moving seawater (Dunstan, 1986, Gehring, 1980) or special aeration techniques. (Swain, 1986)

Table 1 lists ASTM standards pertaining to accelerated testing of coatings.

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APPENDIX C

DATA ON PANEL ID'S AND DRY FILM THICKNESS

CLEVELAND CONDENSATION CABINET, ASTM D 1748

ZINC-RICH

TEST 1A
70 C - 158 F

PANEL ID	DFT
QHA	3.6
QHB	3.5
QHC	3.4
QHD	3.7
QHE	3.3
QHF	3.5
QHG	3.7
QHH	3.5
QHI	3.3
QHJ	4.4
QHK	4.3
QHL	3.4
QHM	3.4
QHN	4.3
QHO	3.9
QHP	4.2
QHQ	3.7
QHR	3.2
QHS	4.4
QHT	2.8
AVE.	3.7
ST. DEV.	0.4

ALKYD

TEST 1B1
70 C - 158 F

PANEL ID	DFT
RQA	4.3
RQB	4.4
RQC	4.2
RQD	4.2
RQE	4.2
RQF	3.7
RQG	4.3
RQH	4.4
RQI	4.3
RQJ	4.5
RQK	4.3
RQL	4.4
RQM	4.3
RQN	3.7
RQO	4.1
RQP	4.1
RQQ	4.0
RQR	4.3
RQS	4.4
RQT	4.4
AVE.	4.2
ST. DEV.	0.2

TEST 1B2
60 C - 140 F

PANEL ID	DFT
RQU	4.3
RQV	4.4
RQW	4.4
RQX	4.1
RQY	4.4
RQZ	4.1
RRR	4.1
RRB	4.2
RRC	3.9
RRD	3.8
RRE	4.1
RRF	4.1
RRG	3.8
RRH	4.2
RRI	4.2
RRJ	4.1
RRK	4.6
RRL	4.4
RRM	4.4
RRN	4.0
AVE.	4.2
ST. DEV.	0.2

TEST 1B3
50 C - 122 F

PANEL ID	DFT
RRO	4.1
RRP	3.7
RRQ	4.7
RRR	4.5
RRS	4.4
RRT	4.3
RRU	4.1
RRV	4.3
RRW	4.0
RRX	4.0
RRY	4.0
RRZ	4.0
RSA	4.1
RSB	4.3
RSC	4.5
RSD	4.0
RSE	4.2
RSF	4.3
RSG	4.6
RSH	4.5
AVE.	4.2
ST. DEV.	0.3

TEST 1B4
40 C - 104 F

PANEL ID	DFT
RPA	4.1
RPB	4.1
RPC	4.2
RPD	4.1
RPE	4.1
RPF	4.1
RPG	3.9
RPH	3.9
RPI	4.3
RPJ	3.9
RPK	4.1
RPL	4.0
RPM	4.1
RPN	4.0
RPO	3.9
RPP	3.8
RPQ	4.2
RPR	4.5
RPS	4.2
RPT	4.1
AVE.	4.1
ST. DEV.	0.1

MARE ISLAND EPOXY 2-COAT

TEST 1C1
70 C - 158 F

PANEL ID	DFT
QUS	3.9
QUT	3.0
QUU	3.3
QUV	3.3
AVE.	3.4
ST. DEV.	0.4

TEST 1C2
60 C - 140 F

PANEL ID	DFT
QRU	3.7
QRV	3.4
QRW	3.8
QRX	3.1
QRY	3.3
QRZ	3.6
QSA	4.3
QSB	3.5
QSC	3.1
QSD	3.6
QSE	3.2
QSF	3.2
QSG	3.4
QSH	4.3
QSI	3.6
QSJ	3.5
QSK	3.7
QSL	3.3
QSM	3.2
QSN	3.3
AVE.	3.5
ST. DEV.	0.3

TEST 1C3
50 C - 122 F

PANEL ID	DFT
QSO	3.8
QSP	3.8
QSQ	4.0
QSR	4.3
QSS	3.6
QST	3.5
QSU	3.5
QSV	3.8
QSW	3.7
QSX	3.9
QSY	3.6
QSZ	3.6
QTA	3.4
QTB	3.8
QTC	3.7
QTD	3.8
QTE	3.7
QTF	4.1
QTG	3.9
QTH	3.7
AVE.	3.8
ST. DEV.	0.2

TEST 1C4
40 C - 104 F

PANEL ID	DFT
QTI	4.1
QTJ	4.3
QTK	4.2
QTL	4.1
QTM	4.3
QTN	4.2
QTO	4.2
QTP	3.7
QTQ	3.2
QTR	3.4
QTS	3.3
QTT	3.9
QTU	3.7
QTV	3.3
QTW	4.0
QTX	3.6
QTY	3.4
QTZ	3.9
QUA	3.4
QUB	3.3
AVE.	3.759
ST. DEV.	0.381

DFT = Dry Film Thickness in mils
AVE. = Average thickness
ST. DEV. = Standard Deviation

DATA ON PANEL ID'S AND DRY FILM THICKNESS (cont.)

HOT WATER IMMERSION

MARE ISLAND EPOXY 2-COAT

TAP WATER

TEST 2A
90 C - 194 F

PANEL ID	FRONT	BACK
QKW	4.0	4.7
QKX	4.6	5.1
QKY	4.6	5.0
QKZ	4.3	4.7
QLA	4.2	5.1
QLB	4.7	5.6
QLC	4.5	5.2
QLD	4.5	5.0
QLE	3.9	4.0
QLF	4.1	4.0
QLG	4.2	4.3
QLH	4.1	4.0
QLI	4.7	5.0
QLJ	4.4	4.7
QLK	4.1	4.9
QLL	3.8	3.9
QLM	4.3	4.2
QLN	4.2	3.9
QLO	4.2	5.3
QLP	4.0	5.4
AVE.	4.3	4.7
ST.DEV.	0.3	0.6

TEST 2B1
90 C - 194 F

PANEL ID	FRONT	BACK
QLS	4.2	4.9
QLT	4.2	5.1
QLU	4.2	5.4
QLV	4.1	5.0
QLW	4.1	5.0
QLX	3.9	5.3
QLY	4.1	5.5
QLZ	3.9	5.1
QMA	4.2	4.7
QMB	4.6	4.9
QMC	4.9	5.3
QMD	4.0	5.5
QME	4.0	5.5
QMF	4.3	5.4
QMG	4.0	5.2
QMH	4.4	5.0
QMI	4.2	5.4
QMJ	3.9	5.1
AVE.	4.2	5.2
ST.DEV.	0.3	0.2

TEST 2B2
80 C - 176 F

PANEL ID	FRONT	BACK
QMK	3.6	4.6
QML	4.1	5.4
QMM	3.7	5.5
QMN	4.2	5.1
QMO	4.1	5.4
QMP	3.4	4.4
QMQ	3.6	4.1
QMR	3.4	4.4
QMS	4.2	4.7
QMT	4.0	4.4
QMU	4.1	4.5
QMV	3.8	4.6
QMW	3.5	4.4
QMX	3.6	4.6
QMY	4.4	4.7
QMZ	4.2	4.7
QNA	4.2	4.4
QNB	4.5	4.5
QNC	3.7	5.1
QND	4.5	5.5
AVE.	3.9	4.7
ST.DEV.	0.3	0.4

TEST 2B3
65 C - 149 F

PANEL ID	FRONT	BACK
QNE	4.1	5.4
QNF	4.2	5.9
QNG	4.0	5.5
QNH	4.3	4.6
QNI	3.9	4.2
QNJ	4.2	4.9
QNK	3.9	5.3
QNL	3.8	5.4
QNM	3.8	5.6
QNN	4.1	5.1
QNO	4.7	4.6
QNP	4.1	4.6
QNQ	4.0	4.4
QNR	4.1	4.3
QNS	4.4	4.5
QNT	4.1	4.2
QNU	4.2	4.6
QNV	3.9	4.6
QNW	3.7	4.1
QNX	4.4	4.7
AVE.	4.1	4.8
ST.DEV.	0.2	0.5

TEST2B4
50 C - 122 F

PANEL ID	FRONT	BACK
REW	3.3	4.2
REX	3.2	4.1
REY	3.7	4.3
REZ	3.7	3.8
RFA	3.5	3.8
RFB	4.1	3.8
RFC	4.1	3.8
RFD	4.4	3.9
RFE	4.0	3.7
RFF	3.4	3.9
RFG	4.2	3.9
RFH	3.5	4.0
RFI	4.0	3.7
RFJ	4.0	4.3
RFK	4.1	4.0
RFL	4.5	4.2
RFM	4.3	3.9
RFN	4.3	3.9
RFO	3.8	4.1
RFP	4.1	4.4
AVE.	3.9	4.0
ST.DEV.	0.4	0.2

DATA ON PANEL ID'S AND DRY FILM THICKNESS (cont.)

PRESSURE/TEMPERATURE IMMERSION

MARE ISLAND EPOXY 2-COAT

MARE ISLAND EPOXY 3-COAT

TEST 3A1
150 psig

PANEL ID	FRONT	BACK
QOS	3.7	4.4
QOT	3.5	4.0
QOU	3.6	3.9
QOV	3.6	4.4
QOW	3.6	4.2
QOX	3.9	4.4
QOY	4.1	4.1
QOZ	3.7	4.2
QPA	3.5	4.6
QPB	3.3	4.3
QPC	3.9	4.1
QPD	3.7	4.2
QPE	3.8	3.9
QPF	3.6	4.3
QPG	3.5	3.9
QPH	3.9	4.5
QPI	3.8	4.2
QPJ	4.0	4.2
QPK	4.3	5.0
QPL	4.4	4.5
AVE.	3.8	4.3
ST. DEV.	0.3	0.3

TEST 3A2
65 psig

PANEL ID	FRONT	BACK
QPM	3.9	4.4
QPN	4.0	4.8
QPO	4.1	4.7
QPP	3.5	4.1
QPQ	4.2	4.3
QPR	3.9	4.5
QPS	3.8	4.3
QPT	3.6	4.3
QPU	3.6	4.2
QPV	3.4	3.8
QPW	4.2	4.9
QPX	4.3	4.8
QPY	3.9	4.1
QPZ	4.0	4.2
QQA	4.0	4.2
QQB	3.7	4.4
QQC	3.7	4.2
QQD	3.9	4.1
QQE	3.8	4.0
QQF	3.4	4.2
QQG	3.8	3.9
QQH	4.2	4.3
QQI	3.6	4.2
QQJ	4.1	4.7
QQK	4.1	4.2
QQL	4.0	4.9
QQM	4.0	4.2
AVE.	3.9	4.3
ST. DEV.	0.2	0.3

TEST 3A3
25 psig

PANEL ID	FRONT	BACK
QQN	4.0	4.1
QOO	4.0	4.6
QQP	4.2	4.5
QQQ	3.5	4.2
QQR	3.6	4.4
QQS	4.0	5.2
QQT	3.6	4.3
QQU	4.0	4.1
QQV	3.4	4.4
QQW	4.6	4.5
QQX	3.6	4.0
QQY	3.6	4.1
QQZ	3.6	4.2
QRA	4.2	4.4
AVE.	3.8	4.4
ST. DEV.	0.4	0.3

TEST 3A4
Atm. pres.

PANEL ID	FRONT	BACK
QUE	3.5	4.0
QUF	3.6	3.8
QUG	3.9	3.8
QUH	3.7	3.9
QUI	3.0	3.8
QUJ	3.2	3.8
QUK	3.4	3.9
QUL	2.9	3.9
QUM	3.5	4.1
QUN	3.1	3.4
QUO	3.9	4.5
QUP	3.4	4.5
QUQ	3.8	5.1
QUR	3.0	3.8
AVE.	3.4	4.0
ST. DEV.	0.3	0.4

TEST 3B1
25psig

PANEL ID	FRONT	BACK
RIE	7.6	7.6
RIF	7.8	7.6
RIG	7.7	7.8
RIH	7.8	8.1
RII	7.9	7.7
RIJ	7.9	7.7
RIK	7.2	6.6
RIL	7.2	6.8
RIM	7.3	6.9
RIN	7.0	6.9
RIO	7.2	7.0
RIP	7.3	7.4
RIQ	7.4	6.7
RIR	7.1	6.9
AVE.	7.5	7.3
ST. DEV.	0.3	0.5

TEST 3B2
Atm. pres.

PANEL ID	FRONT	BACK
QXK	9	8.1
QXL	8.9	8.1
QXM	8.3	8.7
QXN	8.5	8.3
QXO	8	7.8
QXP	8	8.6
QXQ	8.9	7.5
QXR	8.1	8.1
QXS	8.9	8
QXT	8.7	8
QXU	8	8.5
QXV	7.9	8.1
QXW	8.1	8.1
QXX	8.2	8.2
AVE.	8.4	8.2
ST. DEV.	0.4	0.3

DATA ON PANEL ID'S AND DRY FILM THICKNESS (cont.)

SALT/ACETIC ACID FOG

INORGANIC ZINC-RICH (UNTOPCOATED)

MARE ISLAND EPOXY 3-COAT

TEST 4A

TEST 4AA

TEST 4AAA

TEST 4AAS
SCRIBED

TEST 4B

TEST 4BB

TEST 4BS
SCRIBED

PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT
QIO	3.4	QHU	3.2	QJI	3.8	RDY	3.9	QNY	6.9	QUW	7.9	ROQ	7.7		
QIP	3.5	QHV	3.3	QJJ	3.9	RDZ	4.9	QNZ	6.7	QUX	8.7	ROR	7.7		
QIQ	3.1	QHW	3.3	QJK	4.3	REA	5.3	QOA	6.9	QUY	7.7	ROS	7.7		
QIR	4.6	QHX	3.8	QJL	4.3	REB	4.6	QOB	7.2	QUZ	8.3	ROT	6.9		
QIS	4.7	QHY	3.2	QJM	3.4	AVE. 4.7 ST. DEV. 0.6		QOC	7.2	QVA	7.9	AVE. 7.5 ST. DEV. 0.4			
QIT	4.5	QHZ	2.8	QJN	3.4			QOD	6.7	QVB	9.9				
QIU	4.1	QIA	3.3	QJO	4.7	TEST 4AAS SCRIBED		QOE	7.4	QVC	9.0	TEST 4BBS SCRIBED			
QIV	4.6	QIB	3.3	QJP	3.9			QOF	7.4	QVD	8.6				
QIW	4.0	QIC	2.9	QJQ	4.4	PANEL ID	DFT	QOG	7.6	QVE	9.8	PANEL ID	DFT		
QIX	4.6	QID	4.2	QJR	3.8	QJY	3.5	QOH	7.6	QVF	8.7	ROM	7.4		
QIY	4.0	QIE	3.4	QJS	3.1	QJZ	3.1	QOI	7.8	QVG	9.8	RON	7.3		
QIZ	3.6	QIF	3.1	QJT	3.6	QKA	3.1	QOJ	7.2	QVH	9.2	ROO	7.3		
QJA	4.2	QIG	3.5	QJU	2.9	QKB	3.5	QOK	7.1	QVI	8.3	ROP	7.0		
QJB	3.8	QIH	3.5	QJV	3.1	AVE. 3.3 ST. DEV. 0.2		QOL	8.5	QVJ	9.4	AVE. 7.3 ST. DEV. 0.2			
QJC	3.6	QII	3.7	QJW	3.1			QOM	7.4	QVK	9.3				
QJD	3.3	QIJ	3.2	QJX	3.6			QON	6.7	QVL	8.8				
QJE	3.2	QIK	3.8	AVE. 3.7 ST. DEV. 0.5				QOO	7.1	QVM	8.1				
QJF	3.2	QIL	3.3					QOP	7.1	QVN	8.7				
QJG	3.2	QIM	3.4					QOQ	7.3	QVO	8.8				
QJH	4.1	QIN	3.3					QOR	7.4	QVP	7.9				
AVE. 3.9 ST. DEV. 0.5		AVE. 3.4 ST. DEV. 0.3						AVE. 7.3 ST. DEV. 0.4		AVE. 8.7 ST. DEV. 0.7					

DFT = Dry Film Thickness in mils

AVE. = Average thickness

ST. DEV. = Standard Deviation

DATA ON PANEL ID'S AND DRY FILM THICKNESS (cont.)

STEUBENVILLE

KURE BEACH 25 m LOT

ZINC-RICH ZINC + VINYL EPOXY 2-COAT EPOXY 3-COAT

ZINC-RICH ZINC + VINYL EPOXY 2-COAT EPOXY 3-COAT

PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT
QAI	3.7	QCJ	5.1	QDR	4.5	QFC	9.0
QAJ	4.0	QCK	5.0	QDT	4.2	QFD	9.2
QAK	3.6	QCL	5.0	QDU	4.1	QFF	8.7
QAL	3.8	QCM	5.9	QDV	4.6	QFG	8.5
QAM	3.6	QCN	5.0	QDW	4.6	QFH	8.5
QAO	3.6	QCO	5.6	QDX	5.4	QFI	8.3
QAP	3.6	QCP	5.6	QDY	4.4	QFJ	9.2
QAQ	3.7	CCQ	6.7	QDZ	5.1	QFK	9.3
QAR	3.6	QCR	5.3	QEC	4.6	QFM	10.1
QAS	3.8	QCS	7.4	QED	4.7	QFN	9.4
QAT	3.7	QCT	7.5	QEE	4.5	QFO	9.9
QAU	3.8	CCU	7.1	QEF	4.7	QFP	9.1
QAV	3.3	QCV	4.7	QEG	4.7	QFQ	9.2
QAW	3.2	QCW	6.2	QEH	4.3	QFR	9.3
QAX	3.2	CCY	4.6	QEI	4.5	QFS	10.0
QAY	3.5	CCZ	6.6	QEK	4.3	QFT	9.0
QAZ	3.2	QDA	4.6	QEL	4.7	QFU	8.9
QBA	3.7	QDB	4.3	QEM	4.0	QFV	9.8
QBB	3.7	QDC	5.4	QEN	4.6	QFW	9.4
QBC	3.8	QDD	6.3	QEO	4.1	QFX	10.5
QBD	2.9	QDF	6.8	QEP	4.2	QFY	8.8
QBF	2.8	QDH	6.6	QEQ	4.7	QFZ	8.6
QCA	4.0	QDI	7.1	QER	4.1	QGA	9.1
QCC	4.4	QDU	7.8	QES	4.2	QGB	10.9
QCD	4.0	QDK	6.4	QET	4.2	QGC	10.6
QCE	3.7	QDL	7.0	QEU	3.9	QGD	9.3
QCF	4.1	QDM	6.2	QEW	4.8	QGE	9.4
QCG	4.2	QDN	7.7	QEX	4.5	QGF	10.0
QCH	3.7	QDO	7.0	QEY	5.0	QGG	8.8
QCI	4.1	QDP	7.4	QEZ	5.0	QGH	8.1
AVE.	3.7	AVE.	6.1	AVE.	4.5	AVE.	9.3
ST. DEV.	0.4	ST. DEV.	1.1	ST. DEV.	0.3	ST. DEV.	0.7

PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT	PANEL ID	DFT
PVA	4.0	PWJ	6.8	PXR	4.6	PZB	8.6
PVB	2.7	PWK	6.4	PXT	5.0	PZC	9.3
PVC	2.8	PWL	6.8	PXX	5.0	PZD	8.1
PVD	2.9	PWM	6.4	PXY	4.3	PZE	8.5
PVE	3.2	PWN	6.7	PXZ	4.2	PZF	9.0
PVF	3.4	PWP	4.8	PYA	4.2	PZG	8.8
PVG	3.7	PWQ	7.2	PYB	4.7	PZI	8.9
PVH	3.8	PWR	6.9	PYC	5.1	PZJ	7.7
PVI	3.5	PWS	6.8	PYD	4.9	PZK	8.3
PVJ	3.7	PWT	6.8	PYE	4.8	PZL	9.1
PVK	3.8	PWU	6.6	PYF	5.0	PZM	9.2
PVL	2.6	PWV	5.9	PYG	4.5	PZN	9.6
PVM	2.4	PWW	5.1	PYH	5.1	PZP	11.6
PVN	2.9	PWX	6.0	PYI	5.0	PZQ	9.6
PVO	3.7	PWY	6.4	PYJ	4.8	PZS	9.5
PVP	3.7	PWZ	6.2	PYK	4.5	PZT	10.2
PVS	3.7	PXB	5.8	PYL	4.6	PZU	10.1
PVT	3.1	PXC	5.9	PYM	4.6	PZV	10.3
PVU	2.9	PXD	4.9	PYN	4.6	PZW	10.5
PVW	4.0	PXE	6.2	PYO	4.0	PZX	11.2
PVX	4.5	PXF	5.9	PYP	4.3	PZZ	11.2
PVY	2.1	PXG	5.8	PYR	4.2	QAA	10.9
PVZ	3.7	PXH	7.8	PYS	4.5	QAB	9.3
PWA	3.5	PXJ	7.3	PYT	4.7	QAC	8.7
PWB	3.4	PXL	6.8	PYU	4.6	QAD	8.5
PWD	2.0	PXM	7.9	PYV	4.8	QAE	9.0
PWE	2.0	PXN	6.9	PYW	4.9	QAG	8.9
PWG	2.0	PXO	8.0	PYX	4.9	QAH	9.7
PWH	1.9	PXP	7.5	PYY	4.7	QFA	10.0
PWI	3.0	PXQ	7.5	PYZ	4.6	QFB	9.8
AVE.	3.1	AVE.	6.5	AVE.	4.6	AVE.	9.5
ST. DEV.	0.7	ST. DEV.	0.8	ST. DEV.	0.3	ST. DEV.	1.0

APPENDIX D

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